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**DEVELOPMENT AND EVALUATION OF
AN LNG SAMPLING MEASUREMENT SYSTEM**

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Boulder, Colorado 80303

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Prepared for:

LNG Sampling Measurement Supervisory Committee

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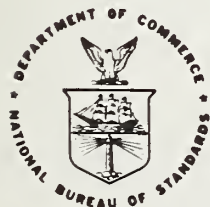
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NBS Interagency Report # 78-887

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DEVELOPMENT AND EVALUATION OF AN LNG SAMPLING MEASUREMENT SYSTEM

W. R. Parrish, J. M. Arvidson and J. F. LaBrecque

This report describes the development and evaluation of procedures and components for sampling and analyzing LNG from flowing streams. Laboratory and field test results showed the total uncertainty in the computed heating value of samples taken with the recommended sampling system could be routinely within ± 0.3 percent; this included the ± 0.1 percent uncertainty in analysis by gas chromatography. Three sample probes and two vaporizer designs were considered. Of the ten operating variables considered, six were found to be important in sampling. Test results were used to establish recommended design and operating criteria.

Key words: Custody transfer; density; gas analysis; gas chromatography; heating value; hydrocarbon; liquefied natural gas; phase equilibrium; pipelines; sampling.

1.0 SUMMARY

Accurate determination of the total dollar value of LNG shipments requires knowing the cargo heating value; this is computed from the composition. At current LNG prices, a composition error of one percent in the heating value would cause an inequity of roughly \$65,000 for one 125,000 m³ LNG shipment. Accurate LNG composition determination requires:

- a) a sample probe for drawing a sample representative of the fluid passing the sampling point,
- b) a sample conditioner for completely vaporizing the sample,
- c) a gas analyzer for accurately and precisely analyzing the sample composition, and
- d) a correct set of operating parameters and procedures.

This report describes the results of a systematic study evaluating all of the above components. The work was confined to the problem of sampling from a flowing LNG stream.

Instead of directly measuring the heating value by gas calorimetry, the gas composition was analyzed so that both heating value and liquid density could be computed.

For analyzing vaporized LNG samples, gas chromatography was selected over mass spectrometry because it was found to be more precise and accurate. By using a gas chromatograph-programmable integrator system, the total uncertainty in the heating value (on a unit volume basis), as computed from the analyzed composition, routinely can be less than ± 0.1 percent. This figure includes a ± 0.03 percent uncertainty in the calibration gas composition; this uncertainty, which neglects the uncertainty of the individual components heating value, is based only on the uncertainty in the weighing process used to prepare the mixtures. The ± 0.1 percent uncertainty also includes a random error of ± 0.06 percent. The random error is three times the estimated standard deviation of ± 0.02 percent. This value represents the maximum value obtained from over 100 measurements of three or more repetitive analyses employing a properly operating gas chromatograph-programmable integrator

system. This report recommends procedures and equipment for analyzing vaporized LNG samples.

Based on the results of sampling tests at the NBS LNG Flow Facility, the total uncertainty of a single measurement in sampling and analyzing LNG mixtures can be less than ± 0.3 percent in the computed heating value. This uncertainty includes the ± 0.03 percent uncertainty in the calibration gas composition plus an uncertainty of ± 0.27 percent for random error. The ± 0.27 percent figure represents three times the upper limit of the standard deviation determined for the combined sampling plus analysis measurement process. The recommended sampling system consists of a side tap probe, a steam or electrically heated tube vaporizer, a gas mixing chamber and a sampling manifold. This report gives recommended sampling system design criteria. Also, it lists the recommended ranges of the operating variables found to affect sampling precision.

Data obtained during a shipboard test has limits of precision of $\pm 0.30\%$. See section 5.2.2. These limits compare very favorably to the $\pm 0.27\%$ obtained on the flow facility.

2.0 INTRODUCTION

Composition plays an important role in determining the total dollar value of LNG shipments. Sampling LNG presents special problems because of possible fractionation during the sampling process. Accurate and precise composition determination of LNG mixtures (and other cryogenic liquid mixtures) requires a Sampling-Measurement System (SMS) which contains:

- a) a sampling probe which draws a sample without altering the composition,
- b) a sample conditioner which completely vaporizes the sample, and
- c) a gas analyzer which accurately and precisely analyzes the sample.

Also, the SMS may include facilities for collecting representative samples of vaporized LNG for transport from the sampling point to the analytical laboratory. The proper operating parameters and procedures must be specified to minimize the total uncertainty in the measurement process.

2.1. Scope of Project

The project consisted of two primary tasks:

- a) the evaluation of vaporized LNG analysis methods, and
- b) the evaluation of LNG sampling techniques. This project carefully

evaluated possible alternatives for each of the components of the SMS. Because of the time limitations only the most promising alternatives were considered in depth. The study covered only sampling from flowing LNG streams. Emphasis was on compositions typical of Algerian LNG (see table 2.1).

2.1.1. Gas Analysis

The initial phase of work evaluated the relative merits of analyzing vaporized LNG samples by gas chromatography (GC) and mass spectrometry (MS); accuracy, precision and simplicity were the primary factors considered. The

Table 2.1. The anticipated composition range of Algerian LNG.
The values in parentheses denote the ranges covered in this report.

Component	Composition Range, mol %
Nitrogen	0.60 to 1.40 (0 to 11)
Methane	84 to 92 (65 to 90)
Ethane	6 to 9.5 (5 to 18)
Propane	2.2 to 3.5 (1.0 to 5.4)
iso Butane	0.3 to 0.5 (0 to 0.8)
normal Butane	0.3 to 0.7 (0.2 to 4.8)
Pentanes and heavier	0 to 0.02 (0 to 0.3)

second phase of work was a study of the effect of various parameters on the accuracy and precision of analysis by GC; the work included an evaluation of GC columns and GC data processors (i.e., integrators). Gas mixtures prepared by weight were used in all of these tests. Also, reliable procedures for obtaining a representative gas sample in a sample cylinder were identified.

2.1.2. LNG Sampling

The sampling portion of the project contained laboratory tests and field confirmation tests; these tests were performed at the NBS LNG Flow Facility and aboard the LNG tanker El Paso Consolidated.

The laboratory tests evaluated three probes -- a pitot tube, a side tap and a reference probe - and two continuous vaporizers - vaporizer A and a reference vaporizer. To distinguish effects due to the vaporizer from those of the probes, the probes were used with a reference vaporizer and vaporizer A with the reference probe. The laboratory evaluation provided a rapid way to determine the effect of a given operating variable while holding other variables constant. The variable's effect upon sampling accuracy was determined by using mixtures of known composition. In addition to the three probes and two vaporizers we considered the following operating variables:

- a) flow rate of the liquid stream,
- b) flow rate through the sampling device,
- c) amount of subcooling (pressure differential between the sample point and the saturated liquid),
- d) temperature of the LNG,
- e) pressure drop upstream of the sample vaporizer,
- f) time-averaging the sample,
- g) vaporizer outlet temperature, and
- h) composition, particularly the effect of pentane and higher hydrocarbons.

The field confirmation tests provided a scale-up test for the conclusions obtained from the laboratory tests. Two series of tests were made in

the NBS flow facility. Here, operating conditions were closer to the conditions of actual LNG facilities in respect to size, pressures and flow rates. However, there was no way to directly determine the correct composition of the mixture in the flow facility. Also, operating conditions were not as constant and well defined as in the laboratory apparatus.

The shipboard tests provided an evaluation of a LNG sampling system under conditions comparable to those in LNG custody transfer operations. The primary variable evaluated in these tests was sampling rate. The actual LNG composition was unknown. However, comparisons were made between liquid samples taken from the inlet header to the ship's vaporizer and gas samples taken from the vaporizer outlet header. This provided a check on the liquid sampler performance.

2.2. Method of Evaluation

In LNG custody transfer, the composition is important because it is needed to compute the heating value and sometimes liquid density. Therefore, in this report we compared experimental results using compositions, computed heating values and computed liquid densities. Several sets [1,2] of ideal gas heating values are available. We chose the values proposed by the A.G.A. [1] (see table 2.2) because, with their method of correcting for gas nonidealities, computed heating values could be directly compared with the results of combustion calorimetry. For convenience, we omitted the non-ideality correction in most of the work. However, in the LNG flow facility tests, the nonideality correction was necessary to make direct comparison with calorimeter measurements. Methods for computing the liquid density are currently under study [3]. For the laboratory tests, we chose the semi-empirical method of Hiza [4] because of its relative simplicity; the extended corresponding states method [3] was used in the field tests.

Table 2.2. Pure component ideal gas heating values [1].

Component	Heating Value ¹ Btu/scf	MJ/m ³
N ₂	0	0
CH ₄	1012.1	37.701
C ₂ H ₆	1773.0	66.045
C ₃ H ₈	2523.3	93.994
iC ₄ H ₁₀	3260.7	121.46
nC ₄ H ₁₀	3269.8	121.80
iC ₅ H ₁₂	4008.7	149.33
nC ₅ H ₁₂	4018.9	149.71
C ₆ H ₁₄ , average	4764.4	177.48
C ₇ H ₁₆ , average	5509.7	205.34

¹The ideal gas volumetric heating value is based on the standard conditions of 60°F (15.6°C) and 30 inches of mercury (0.1016 MPa).

This report makes extensive use of statistical techniques in evaluating the experimental data. Table 2.3 lists the definitions of the statistical terms used.

Table 2.3. Definition of statistical terms used in this report.

Accuracy	- the closeness to the true value.
Average value ($\bar{x}$)	- the arithmetic average of replicate measurements. The average value represents the most likely value of the mean.
Bias (B)	- a systematic offset between the mean and true value.
Estimated standard deviation (s)	- an estimate, based on a finite number of replicate measurements, of the standard deviation of a measurement process.
Mean (X)	- a value, usually unknown, about which the measured values of replicate measurements randomly scatter.
Measurement process	- a series of steps used to assign a numerical value to a physical property.
Random error	- an error related only to the random behavior within a measurement process, i.e., not a systematic offset.
Replicate measurements	- two or more measurements made under conditions where all controllable variables are identical for all measurements.
Standard deviation (σ)	- a parameter often used as a measure of the scatter of random error in a measurement process.
Systematic error	- an estimate of the upper limit of the unknown but fixed errors of a measurement process.
Total uncertainty	- the estimated maximum error in a measurement process. In this report it is computed by summing the systematic error and the upper limit of the random error -- taken to be three times the estimated standard deviation.
Confidence interval	- an interval, based on replicate measurements, for a parameter which will include the true value of the parameter a specified percentage of the time.
Precision	- the closeness with which the results of replicate measurements agree -- usually quantified by the estimated standard deviation.

Determining the composition, like all measurement processes, involves experimental error. This error may be considered in terms of the accuracy of a process (bias) and its precision. Bias of a measurement process represents how closely the mean of the measured quantity conforms to the true value. Precision represents how closely the values obtained from a number of independent, repeated measurements agree, i.e., random error.

Replicate measurements yield estimates of both bias and precision. The difference between the average value, $\bar{x}$, of the measured values, x_i , and the true value is an estimate of the bias in the measurement process. Scatter about the average value is an estimate of the precision; normally the scatter is quantitated in terms of the estimated standard deviation, s. In this report we assume that the random errors follow the statistics of a normal distribution. The estimated standard deviation is given by

$$s = \sqrt{\frac{n}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2} \quad (2.1)$$

where n denotes the number of replicate measurements.

The values of $\bar{x}$ and s will vary among different sets of the same measurement. However, they will vary about the true mean, X , and standard deviation σ . The values of X and σ are considered known only after making a very large number of measurements. The values of X and σ are estimated by using $\bar{x}$, s and confidence intervals; for example, a 99 percent confidence interval for the mean value signifies that 99 percent of the time the interval will include the true value. In this case the interval is given by

$$\bar{x} - t \cdot s / \sqrt{n} < X < \bar{x} + t \cdot s / \sqrt{n} \quad (2.2)$$

where t is the Student's statistic for a 99 percent confidence of n repeated measurements.

For the standard deviation, the confidence interval is given by

$$B_L \cdot s < \sigma < B_U \cdot s \quad (2.3)$$

where B_L and B_U are values derived from the Chi Squared statistic; they are functions of s , n and the degree of confidence (e.g., 99 percent). The interval in equation (2.3) is unsymmetrical about σ . In this report we present values of n , $\bar{x}$ and s and, as a first approximation assume that $\bar{x}$ and s are equal to X and σ .

We use the concept of total uncertainty in assuming a limit on the error in a measurement process. Total uncertainty reflects two errors -- systematic and random. The systematic error represents an estimate of the upper limit of the unknown, nonrandom errors of the measurement process. Its estimate is based on understanding the nonrandom errors of the components within the measurement process. The random error is estimated to be three times the estimated standard deviation - this roughly corresponds to a 99.7 percent confidence interval (it would equal a 99.7 percent confidence interval if the estimated standard deviation equaled the standard deviation).

One of the major goals of this project was to determine which operating variables affected the precision of composition measurement. To determine if a given variable changed the precision, measurements would be made at two (or more) values of the variable. How confidently we could detect the change in precision depended upon the number of samples taken under each set of conditions and upon the observed change in the estimated standard deviation. As shown in table 2.4, we have a 65 percent chance of detecting a difference in the precision when there is a factor of four difference between the observed standard deviation and when the measurement is repeated four times with each value of the operating variable; for a 62 percent chance of detecting a difference when there is a factor of two difference in the observed standard deviation it would take 10 measurements with each value of the operating variable.

Table 2.4. Probability of detecting a difference in the standard deviation between two sets of conditions (A and B) when the measurements have a factor λ difference in standard deviations and they are repeated N times under each set of conditions [5].

N	Probability, percent			
	$\lambda (= \sigma_B/\sigma_A)$			
	1	2	3	4
4	5	25	49	65
6	5	41	71	87
8	5	52	87	95
10	5	62	93	99

3.0 PREVIOUS WORK

To our knowledge no systematic evaluation of LNG Sampling Measurement Systems (SMS) has been made. However, there are publications on gas analysis methods and on LNG sampling techniques.

3.1. Gas Analysis

We are unaware of any publications comparing gas chromatographic (GC) and mass spectrometric (MS) analyses of natural gas. However, ASTM standards exist for natural gas analysis by gas chromatography [6] and mass spectrometry [7]. Each standard gives the estimated reproducibility of analysis; values are given for the case of one laboratory and apparatus, and for the case of different laboratories. The ASTM standards indicate that the analyses should be considered suspect if differences between replicate analyses are greater than those given in table 3.1; also, the table gives the estimated error in heating values for these differences assuming the composition range given in table 2.1.

There are several papers discussing different GC column configurations; these are discussed in section 4.

3.2. LNG Sampling

Dalmaze [8] and Trigo [9] discussed two similar continuous SMS which are used in Europe. Figure 3.1 is a schematic of Dalmaze's system. Both use an upstream facing pitot tube located at the LNG pipe centerline as the probe. To prevent premature vaporization the probe line is cooled by an outer jacket through which LNG is circulated and returned to the main line. The sample then flows through a check valve to prevent backflashing and past an electrically heated vaporizer. Trigo's system replaces the check valve with a filter. The vaporized sample goes through a vessel to dampen pressure pulsations (if operating at less than 30 psig (0.3 MPa)), past a pressure controller, and to a chromatograph. Neither author describes the details of the gas analysis. Dalmaze's data showed a precision of ± 0.6 percent in the computed heating value of liquid samples taken upstream of a LNG vaporizer;

Table 3.1. ASTM [6,7] estimates of reproducibility between replicate natural gas analyses, and the resulting percentage error in heating value for the anticipated Algerian LNG compositions given in table 2.1., using the gas chromatograph (GC) and the mass spectrometer (MS).

Component	Reproducibility between analyses by							
	Same Operator and Apparatus				Different Operators and Apparatus			
	GC		MS		GC		MS	
	mol %	% error in heating value	mol %	% error in heating value	mol %	% error in heating value	mol %	% error in heating value
N ₂	0.03	0.07	0.2	0.4	0.06	0.1	0.3	0.7
CH ₄	0.3	0.4	0.2	0.3	0.6	0.8	0.5	0.6
C ₂ H ₆	0.15	0.07	0.1	0.05	0.20	0.1	0.3	0.1
C ₃ H ₈	0.05	0.01	0.02	0.01	0.1	0.02	0.06	0.02
iC ₄ H ₁₀	0.03	0.03	--	--	0.06	0.06	--	--
nC ₄ H ₁₀	0.03	0.03	--	--	0.06	0.06	--	--
C ₅ ¹	0.03	0.04 ¹	--	--	0.06	0.04 ¹	--	--

¹Error in C₅⁺ limited to 0.02 mol % which is the maximum anticipated composition.

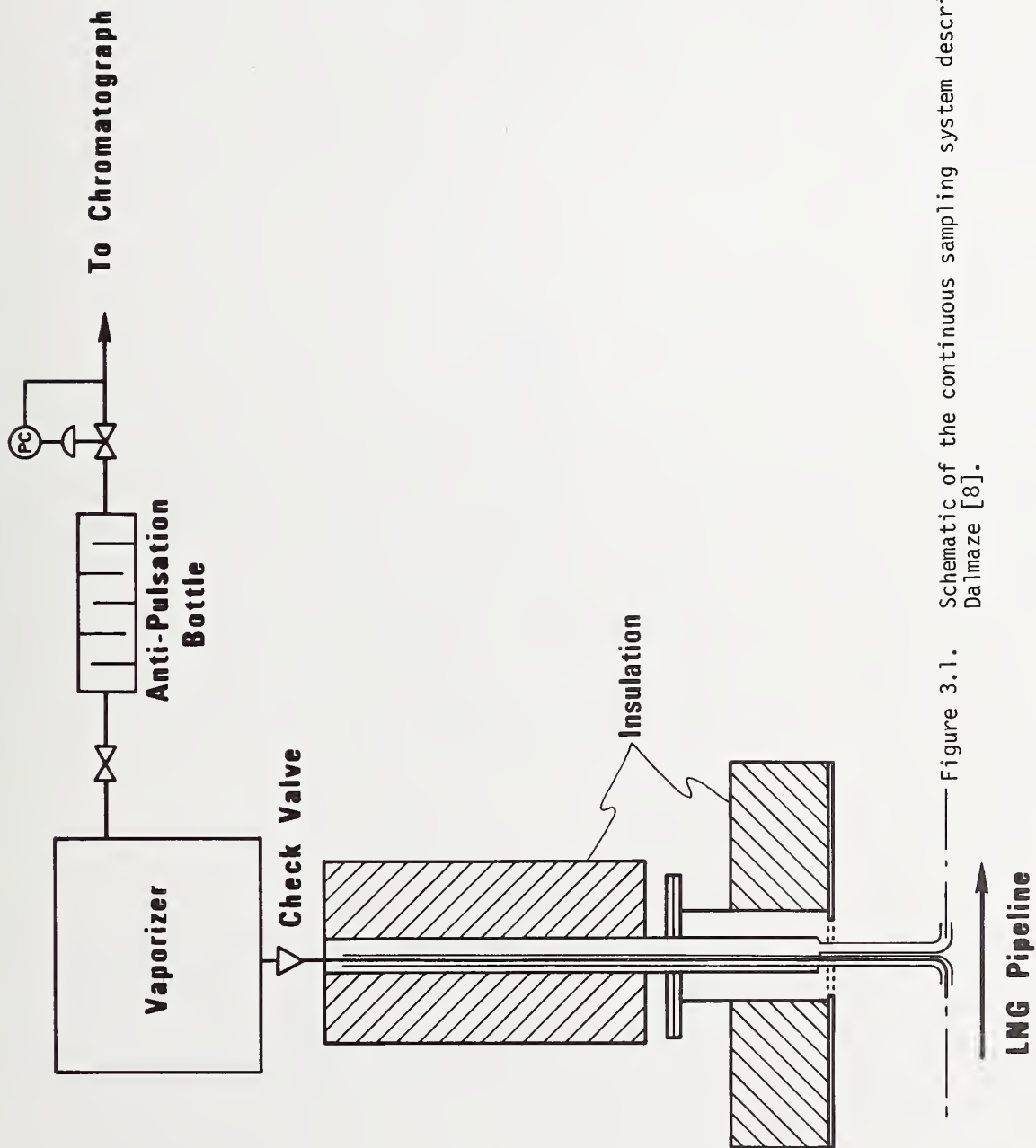


Figure 3.1. Schematic of the continuous sampling system described by Dalmaze [8].

by sampling the totally vaporized LNG stream the precision was 0.4 percent. (The precision is defined here as three times the estimated standard deviation.) The difference in the average computed heating values between the two sampling methods is about 0.01 percent when 30 sets of samples were averaged over seven hours. Dalmazé reports that this difference is well within the error due to gas analysis alone.

There are at least two batch sampling devices which have been tried. One is the cryogenic sampler [10] shown in figure 3.2. LNG passes through a cooling passage which surrounds a cup contained in an evacuated outer container. Once the cup is at LNG temperature the sample valve is opened and the cup is filled with liquid. After the cup is full the sample valve is closed, the sample lines are disconnected and the sampler is allowed to warm to ambient temperature. The ratio of the cup volume to the total chamber volume is such that the final pressure will be approximately 500 psig (3.6 MPa) for LNG. A valve at the bottom of the sampler is used to draw a gas sample for analysis.

The other batch sampling device which collects the liquid is described by Cook [11] and is shown in figure 3.3. To take a sample, both valves are opened to allow LNG to flow through the sampler which is well insulated. Once the sampler is at LNG temperature, as evidenced by a continuous liquid flow from the device, both valves are closed. Then the device is removed from the sampling line and allowed to warm up. After the initial pressure rise, a pressure relief valve which is set above the cricondenbar (the maximum pressure at which a mixture may exist in two phases) of the mixture (> 2000 psi (13.8 MPa)) keeps the sample at a constant pressure and in one phase. Once the sampler is warm it can be connected directly to an analytical instrument.

Methods for collecting a gas sample from a pipeline have been discussed by Miller [12]; also, an ASTM standard exists for natural gas sampling from pipelines [13]. Miller concludes that all of the following filling methods give good results:

- a) liquid displacement,
- b) purge by fill and empty,
- c) purge at a constant flow rate, and
- d) fill of evacuated cylinder with no purge.

Also, he briefly describes the two batch sampling devices used for collecting LNG as a liquid which were described above.

Finally, Chapman and Payne [14] give some general guidelines for sampling flowing streams. Although they do not describe cryogenic sampling systems, many of their guidelines still apply.

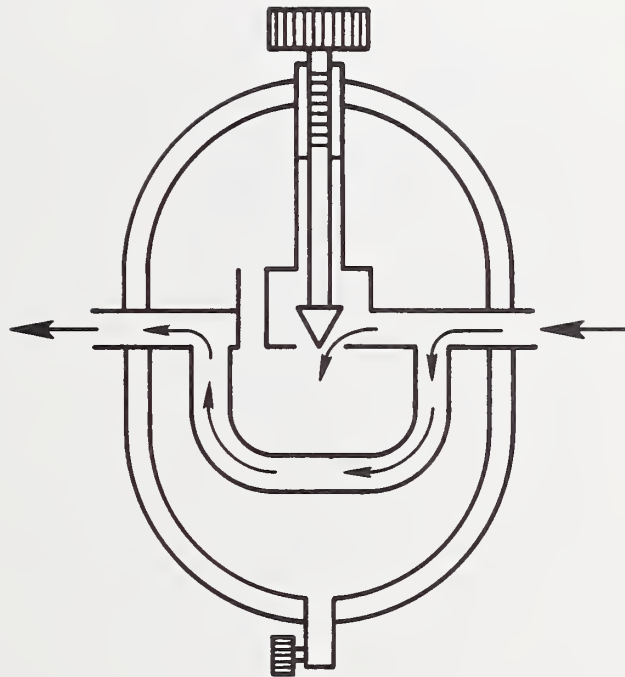


Figure 3.2. Schematic of cryogenic sampler [10].

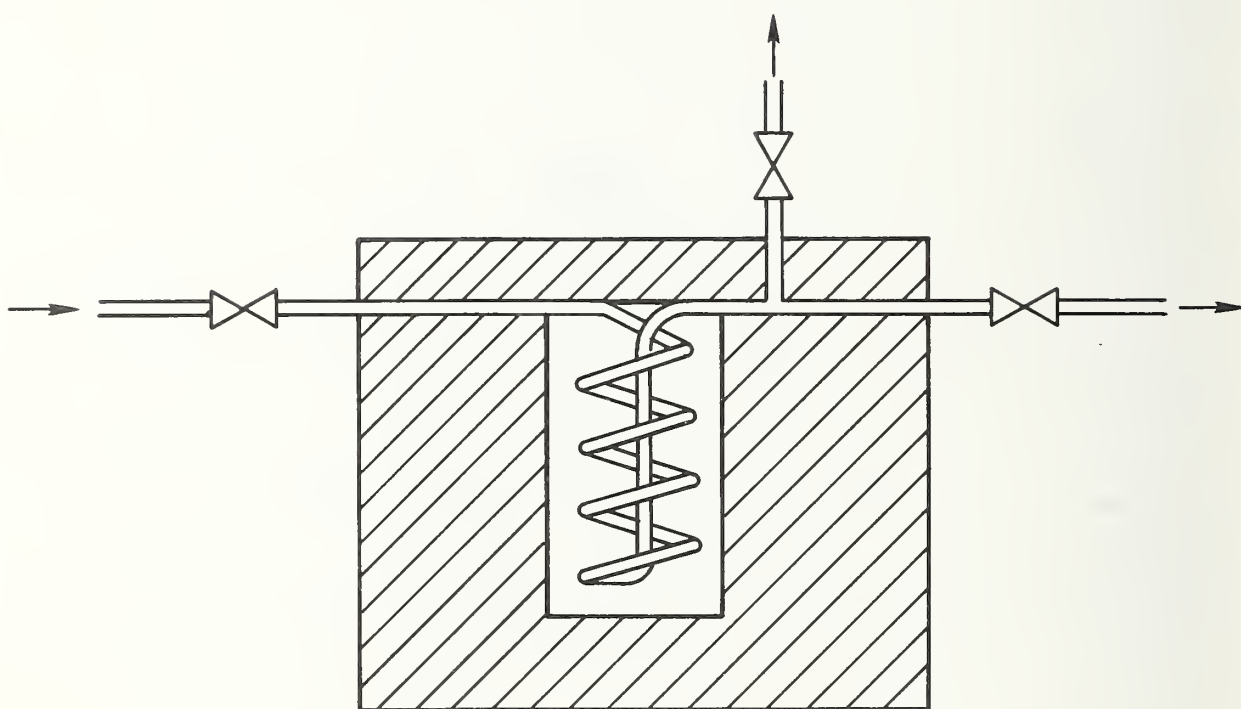


Figure 3.3. Schematic of LNG sampler described by Cook [11].

4.0 GAS ANALYSIS

Before any sampling data could be taken we had to determine the uncertainty involved in gas analysis. This evaluation and a comparison between gas chromatography (GC) and mass spectrometry (MS) were made early in the project. Later, we more carefully evaluated the effect of various operating variables, column configurations, and digital integrators on analysis precision and accuracy. Appendix 7.1 lists all of the data obtained for the gas analysis tests.

4.1. Comparison Between Gas Chromatography and Mass Spectrometry

A comparison of precision and accuracy of GC and mass spectrometric analysis of LNG type mixtures was made by having a local MS laboratory analyze some prepared mixtures; these mixtures also were analyzed using a GC in our laboratory. The MS laboratory was capable of general quantitative analysis, including hydrocarbon analyses, on a routine basis.

4.1.1. Gas Chromatograph System

4.1.1.1. Apparatus

In this phase of the work we used a gas chromatograph containing two columns and a filament-type thermal conductivity detector, in conjunction with a programmable, digital integrator (integrator A). Traditionally, whenever feasible, peak heights were preferred over peak areas for computing compositions from GC analyses. The reasons for this were that peak heights were easier to measure and they were oftentimes more precise than the mechanical devices available for measuring peak areas. However, programmable integrators eliminate the drawbacks of using peak areas. Several sets of calibrations and analyses were made using both peak heights and peak areas. (Integrator A reports peak areas and/or peak heights.) These tests showed that the compositions based on peak heights had an estimated standard deviation which was four to five times greater than the estimated standard deviations for the the compositions computed from peak areas. Based on these results and the fact that the linear response between peak height and composition has a limited range, we used peak areas throughout this work.

Table 4.1 lists the GC operating conditions. Figure 4.1 is a schematic of the GC and ancillary equipment while figure 4.2 shows a chromatogram (on a logarithmic scale) for a gas mixture typical of Algerian LNG. The GC contained a sample valve which was used to inject the gas sample from either of two sample loops into the carrier gas stream. Helium carrier gas was maintained at a constant flow by using two stages of pressure regulation. The column bypass valve was used to bypass Column 2. In the bypass mode the carrier gas passed through a needle valve set to have the same pressure drop as Column 2; this minimized the baseline upset during valve switching.

Table 4.1. Gas chromatograph operating conditions.

Carrier gas flow rate	27 cc/min (at 14.7 psia (0.1 MPa))
Sample loop volumes	$\sim 0.1 \text{ cm}^3$ each
Sample loop temperature	90°C
Column 1 temperature	25°C
(10 ft (3.0 m) x 1/8 in (3.2 mm) of Durapak*, 60/80 mesh)	
Column 2 temperature	25°C
(6 ft (1.8 m) x 1/8 in (3.2 mm) of Porapak Q, 60/80 mesh)	
Detector temperature	120°C
Filament current	220 ma

* Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

The integrator had a variety of operating parameters and timed events which could be used to maximize the precision of the data reduction of a chromatogram. The use of these parameters is illustrated in the next section.

4.1.1.2. Calibration procedure

All GC's require calibration with one or more mixtures of known composition; these mixtures should have a composition similar to that of the unknown mixtures. In this work we prepared all of the calibration mixtures by weight using high purity components; table 4.2 lists the component concentrations and their estimated uncertainties for all the prepared mixtures used here.

To make a calibration run, the sample loop first was purged by alternately evacuating and filling with the calibration gas. We found that one to three purges were adequate provided that the manifolding was arranged so that all of the gas passed through the sample loop during purging (see figure 4.1). However, it was imperative that all connections were leak-tight. After purging, the sample loop was filled to the desired pressure -- in our case 2 psig (0.11 MPa) -- and the sample valve was switched, injecting the mixture into the carrier gas stream. During the first three minutes of the run Column 2 remained in series; this allowed the nitrogen and methane to travel through both columns and the detector. (These columns did not resolve oxygen and nitrogen; therefore, any oxygen present was included in the nitrogen peak.) Once the methane was out of Column 2, the column was bypassed, trapping the ethane. The integrator made a "forced baseline" just prior to the valve switch and ignored the tailing baseline after the switch. (The dotted lines under each peak in figure 4.2 represent the baseline as determined by the integrator.) The integrator then picked the bottom of the baseline drift as the baseline for the propane peak. Column 2 remained bypassed until the butanes passed through Column 1. At 9 minutes Column 2 was switched in again, allowing ethane to elute from the column. As before, the integrator

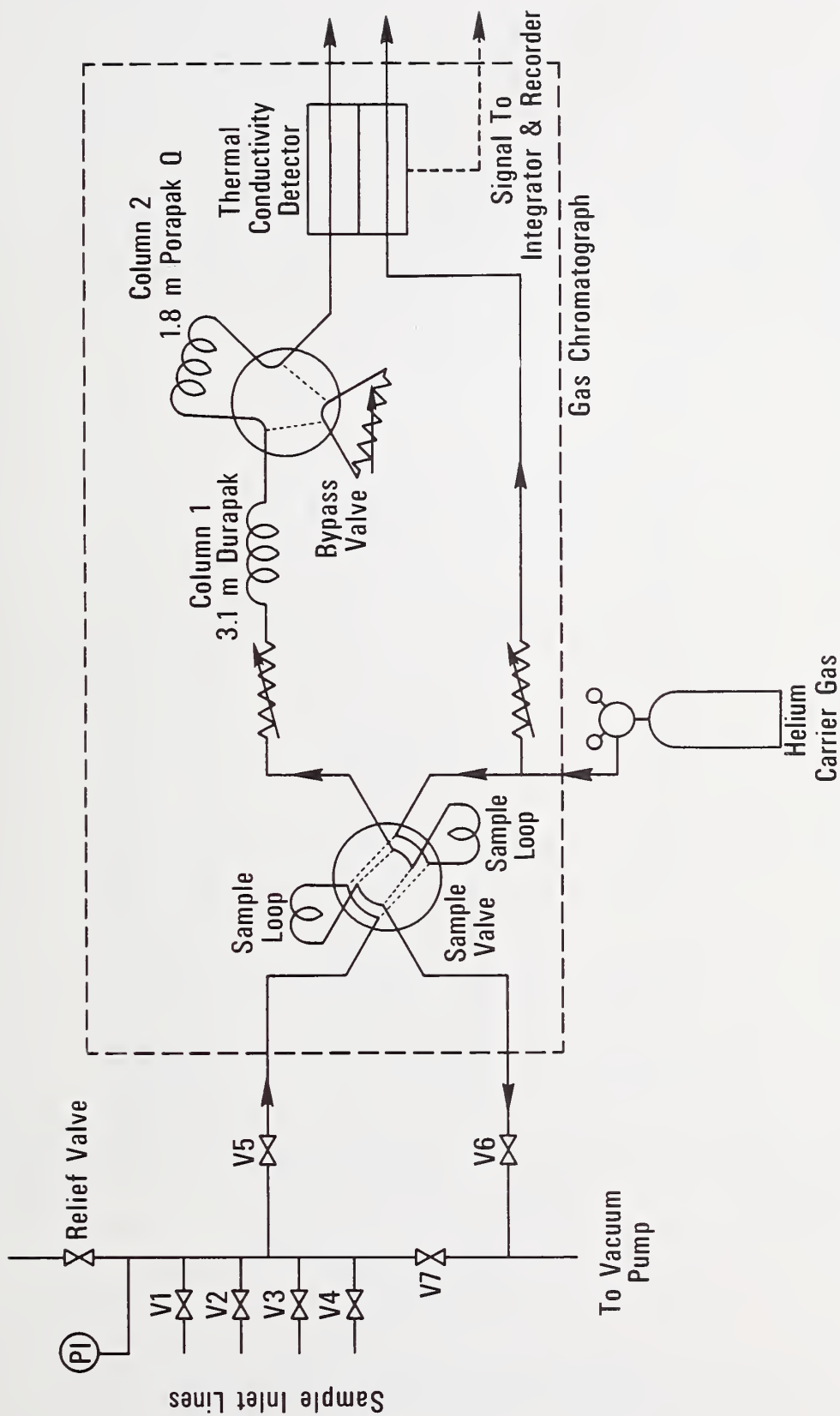


Figure 4.1. Flow schematic of two column gas chromatography system.

Component	C_2H_6	nC_4H_{10}	iC_4H_{10}	C_3H_8	CH_4	N_2
Time, min.	12.74	7.94	6.72	3.63	2.44	2.05
Area, Arib. units	70890	8643	6142	30721	480864	9108
Peak Height, mV	2.846	0.542	0.461	4.095	82.784	2.087
Concentration, mol%	5.477	0.707	0.519	2.980	85.936	1.382

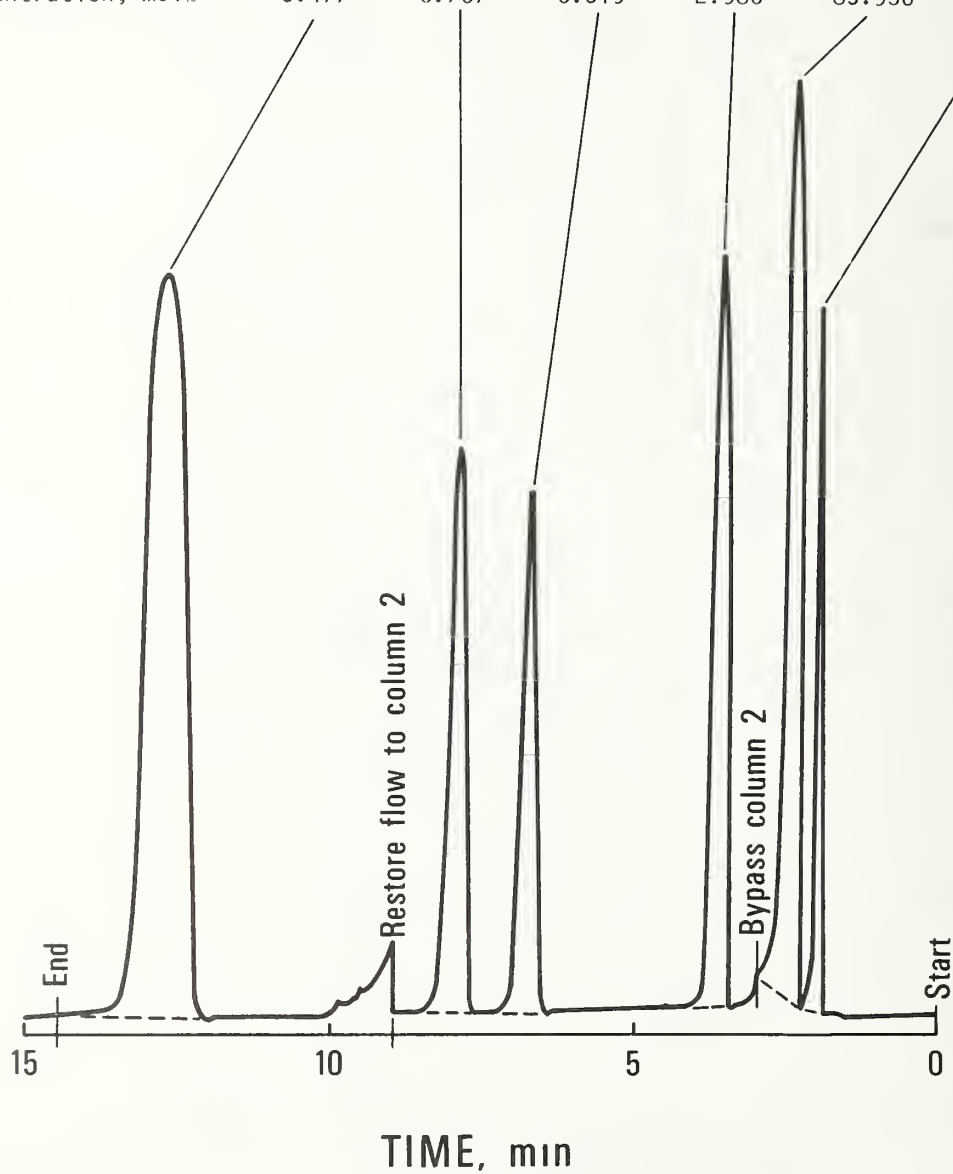


Figure 4.2. A chromatogram of a natural gas mixture. The peak heights are shown on a logarithmic scale.

Table 4.2. Compositions, ideal gas heating values and their estimated uncertainty¹ for all mixtures, prepared by weight, used in this work.

Mixture	Composition, mol %								Heating Value ²	
	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	Btu/scf	MJ/m ³
A	4.316 +0.005 _	81.249 +0.008 _	4.746 +0.005 _	4.866 +0.001 _	2.409 +0.002 _	2.413 +0.002 _			1186.7 +0.3 _	44.205 +0.011 _
B	1.349 +0.002 _	88.118 +0.003 _	8.392 +0.002 _	0.978 +0.001 _	0.481 +0.0004 _	0.682 +0.0004 _			1103.3 +0.1 _	41.098 +0.004 _
C	0.601 +0.002 _	90.622 +0.002 _	6.027 +0.002 _	2.154 +0.001 _	0.300 +0.0004 _	0.306 +0.0004 _			1098.2 +0.1 _	40.909 +0.004 _
D	0	85.317 +0.008 _	5.077 +0.004 _	4.855 +0.001 _	0	4.751 +0.002 _			1231.4 +0.2 _	45.870 +0.007 _
E	1.382 +0.002 _	85.936 +0.003 _	8.477 +0.002 _	2.980 +0.001 _	0.519 +0.0004 _	0.707 +0.0004 _			1135.3 +0.1 _	42.290 +0.004 _
F	0.973 +0.002 _	88.225 +0.003 _	7.259 +0.002 _	2.561 +0.001 _	0.490 +0.0004 _	0.492 +0.0004 _			1118.3 +0.1 _	41.657 +0.004 _
G	11.090 +0.002 _	65.732 +0.003 _	16.283 +0.002 _	5.443 +0.001 _	0.490 +0.0004 _	0.963 +0.0004 _			1138.8 +0.1 _	42.421 +0.004 _
H	0.599 +0.0004 _	90.070 +0.0007 _	6.537 +0.0004 _	2.200 +0.0001 _	0.2912 +0.0002 _	0.2837 +0.0002 _	0.0101 +0.0002 _	0.0113 +0.0002 _	1102.7 +0.03 _	41.075 +0.001 _

¹The uncertainty is based only on the error in the weight measurement; details of this error analysis are presented in [31].

²The ideal gas heating value is computed as the sum of the mol fraction of each component times its ideal gas heating value [1] at standard conditions. The uncertainty in the heating value is computed as the sum of uncertainties in the mol fraction of each component times its ideal gas heating value.

ignored the baseline upset due to valve switching. The tailing after switching valves was caused by a temporary decrease in carrier gas flow as pressure gradients in the line were being reestablished. The two peaks on the second tail were caused by minute air leakage during the valve switch. This leakage is caused by diffusion and can occur in pressurized helium and hydrogen systems.

The total analysis time was 14.5 minutes. If pentanes were present the same procedure was used but the analysis time increased to 24 minutes. The prepared mixtures in this study did not contain hexanes.

Using the areas under each peak and the known composition of the calibration gas, relative response factors were computed for each component. Normally, two or three calibration runs were made and the response factors averaged for each component. The GC was calibrated at the beginning of each operating day.

4.1.1.3. Analysis procedure

The procedure for an analysis of an unknown mixture was the same as a calibration run. However, in this case the relative response factors were used with the peak areas to determine the composition. In our work we normalized the results; this eliminated the need to know the exact amount of sample injected. However, we tried to keep the sample amount, i.e., sample pressure, constant to within $\pm 5\%$ because variations in sample size may cause subtle effects which could diminish the analytical accuracy (see section 4.4).

4.1.2. Mass Spectrometer System

4.1.2.1. Apparatus

The mass spectrometer used for this work was a 180° sector, high precision gas mass spectrometer [15]. It electronically scanned a mass range of two to 200 with the output recorded on a strip chart. By adjusting the sample pressure the pressure at the controlled leak could be set between 1 to 100 microns (0.1 to 13 Pa). The controlled leak permitted a small amount of sample to continuously enter the ion chamber; the leak rate was proportional to the pressure upstream of the controlled leak.

4.1.2.2. Calibration procedure

Prior to running a calibration (or analysis), a background scan was made by closing the valve upstream of the controlled leak. Any peaks from the residual gas were noted. Next, high purity hydrocarbons (> 99.99 percent) were injected, one at a time, at pressures comparable to the expected partial pressures in the unknown mixture. The MS response was slightly non linear; however, this effect was negligible as long as the calibration pressure was within 3 to 4 microns (0.4 to 0.5 Pa) of the components partial pressure in the unknown mixture.

Hydrocarbons fracture inside the ion chamber. Each hydrocarbon has a characteristic cracking pattern and at least one mass peak which is unique to that hydrocarbon. The response factor was computed for each component by dividing the characteristic peak height (corrected for background) by the known calibration gas pressure. (Resolving the iso and normal butane required solving two simultaneous equations.)

4.1.2.3. Analysis procedure

After evacuating the line connecting the sample cylinder to the MS, the sample pressure was reduced to the desired level by a series of expansions into evacuated volumes. Then, the sample was opened to the controlled leak and a mass scan was made. Using response factors, peak heights (corrected for background) and sample pressure, the composition of each component was calculated.

4.1.3. Evaluation of Results

The MS laboratory received two sets of six samples. The first set contained duplicate samples of mixtures A, B and D, while the second set contained duplicate samples of mixtures B, C and D; the samples were labeled with the approximate composition of the sample. The MS lab reported two analyses on each sample. Table 4.3 gives the results of the MS and GC analyses. The raw data are listed in Appendix 7.1. Since this was not a statistically balanced test, only a comparison of the bias (i.e., accuracy) and precision could be made; there was no way to determine what factors affected the accuracy and precision. However, it was found that the second set of MS analyses showed a significant improvement in the standard deviation, whereas, there was little change in the bias. In all cases both biases and standard deviations for the GC analyses were much smaller than those of the MS analyses.

The manager of the analytical laboratory where the MS analyses were made feels that the MS is an important qualitative and quantitative tool [15]. However, for routine gas analyses, where all of the components are known, he believes that a properly designed and operated GC-digital integrator system will be more precise than the MS. (His laboratory contains several types of both MS and GC instruments.)

The precision of the MS analysis results obtained in our study is poorer than would be expected based on the ASTM standard [7] (see table 3.1). One possible reason is that the reference mixture used in the ASTM standard to determine analytical precision did not include butanes; butanes and heavier hydrocarbons made the data reduction of the mass spectrum much more difficult. Also, the concentration levels of components other than methane were lower in the ASTM mixture than in our study.

In addition to our comparison we received a tabulation of roughly 30 sets of analyses of natural gas mixtures containing 95 mol percent or more

Table 4.3. Estimated biases and standard deviations in the composition and ideal gas heating value from gas chromatographic (GC) and mass spectrometric (MS) analyses.

Mixture	Analysis Method	Number of Analyses	Estimated Bias ¹ (B) and Standard Deviation (s) in Composition, mol %																Heating Value Btu/scf (MJ/m ³)
			N ₂						CH ₄		C ₂ H ₆		C ₃ H ₈		iC ₄ H ₁₀		nC ₄ H ₁₀		
			B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s	
A	MS	4	3.49 + 1.24	0.78	-4.44 + 1.11	0.70	-0.24 + 0.38	0.24	-- ⁴	0.62	--	0.21	-0.44 + 0.03	0.022	-37.3 + 9.2 - 1.39 + 0.34	5.8 (0.216)			
B	GC	6	--	0.20	--	0.27	--	0.011	--	0.021	--	0.020	--	0.036	1.38 + 1.13 - 0.051 + 0.042	0.91 (0.034)			
	MS	8	--	0.37 ²	--	0.23 ²	-	0.21 ²	--	0.070 ²	--	0.042 ²	--	0.015 ²	--	5.0 ² (0.19)			
C	GC	14	--	0.045	--	0.044	--	0.0184	--	0.004	--	0.005	--	0.005	--	0.50 (0.019)			
	MS	4	--	1.57	--	1.59	--	0.25	-0.94 + 0.78	0.49	0.31 + 0.14	0.088	-0.096 + 0.013	0.082	-31.3 + 18.0 - 1.16 + 0.67	11.3 (0.42)			
D	GC	13	0.030 + 0.019	0.031	-0.068 + 0.025	0.040	0.016 + 0.007	0.011	--	0.013	--	0.003	--	0.002	--	0.58 (0.022)			
	MS	8	--	0.21 ²	--	0.29 ²	--	0.22 ²	--	0.42 ²	--	0.029 ²	--	0.033 ²	--	8.3 ² (0.31)			
E ³	GC	8	0.009 + 0.001	0.001	--	0.053	--	0.012	--	0.009	--	--	--	0.038	--	1.04 (0.039)			
	GC	3	--	0.008	--	0.061	--	0.039	--	0.027	--	0.005	--	0.008	--	0.95 (0.035)			

methane. Samples of each gas mixture were analyzed by MS in two different industrial laboratories and by GC. Also, the heating value of the mixture was measured by using combustion calorimetry. Table 4.4 lists the average error and its estimated standard deviation between the computed heating values, based on gas analysis, and the measured values. Clearly, the GC analysis is much better than one MS laboratory and has a better estimated standard deviation than the second MS laboratory.

Table 4.4. The average error and its estimated standard deviation between the heating values computed from analyses by gas chromatography (GC), two different mass spectrometers (MS) and the heating value as measured by combustion calorimetry for natural gas mixtures containing 95 mol % or more methane [16].

Analytical Method	Average Error ¹ Btu/scf (MJ/m ³)	Standard Deviation Btu/scf (MJ/m ³)
GC	1.27 (0.0473)	0.55 (0.0205)
MS I	5.05 (0.1881)	3.63 (0.1352)
MS II	1.17 (0.0436)	1.46 (0.0544)

¹The average error is defined here as the average of the absolute value of the difference between the computed and measured heating values.

4.2. Statistical Evaluation of GC Analyses

Based on the above results the GC was selected to be used in this project. Therefore, a statistically defined test was required to determine the analytical precision and accuracy of the GC; also, the effect of various operating parameters needed to be defined. In this test we looked for operator, composition, and day-to-day effects. We used two operators, four mixtures (mixtures C, E, F and G, table 4.2) and ran the tests for four days. Figure 4.3 shows the computed ideal gas heating values for mixture E (the calibration gas) as a function of chronological order and operator; the σ error bar represents one estimated standard deviation (0.20 percent) in the heating value. Although there was no operator dependence, there was a day to day shift in the average value; this probably was due to calibration shifts. Also, there were apparent changes in the scatter from day to day which could not be explained; however, excluding the outlier on the last day, the total scatter was 0.09%.

The outlying point on the fourth day illustrates an instrument malfunction discovered in this test. Roughly 20% of the analyses contained one component, usually nitrogen, methane or ethane, whose composition was more than four estimated standard deviations from the average value. During this period, the integrator started malfunctioning and had to be returned to the factory. After receiving the repaired instrument another test was performed,

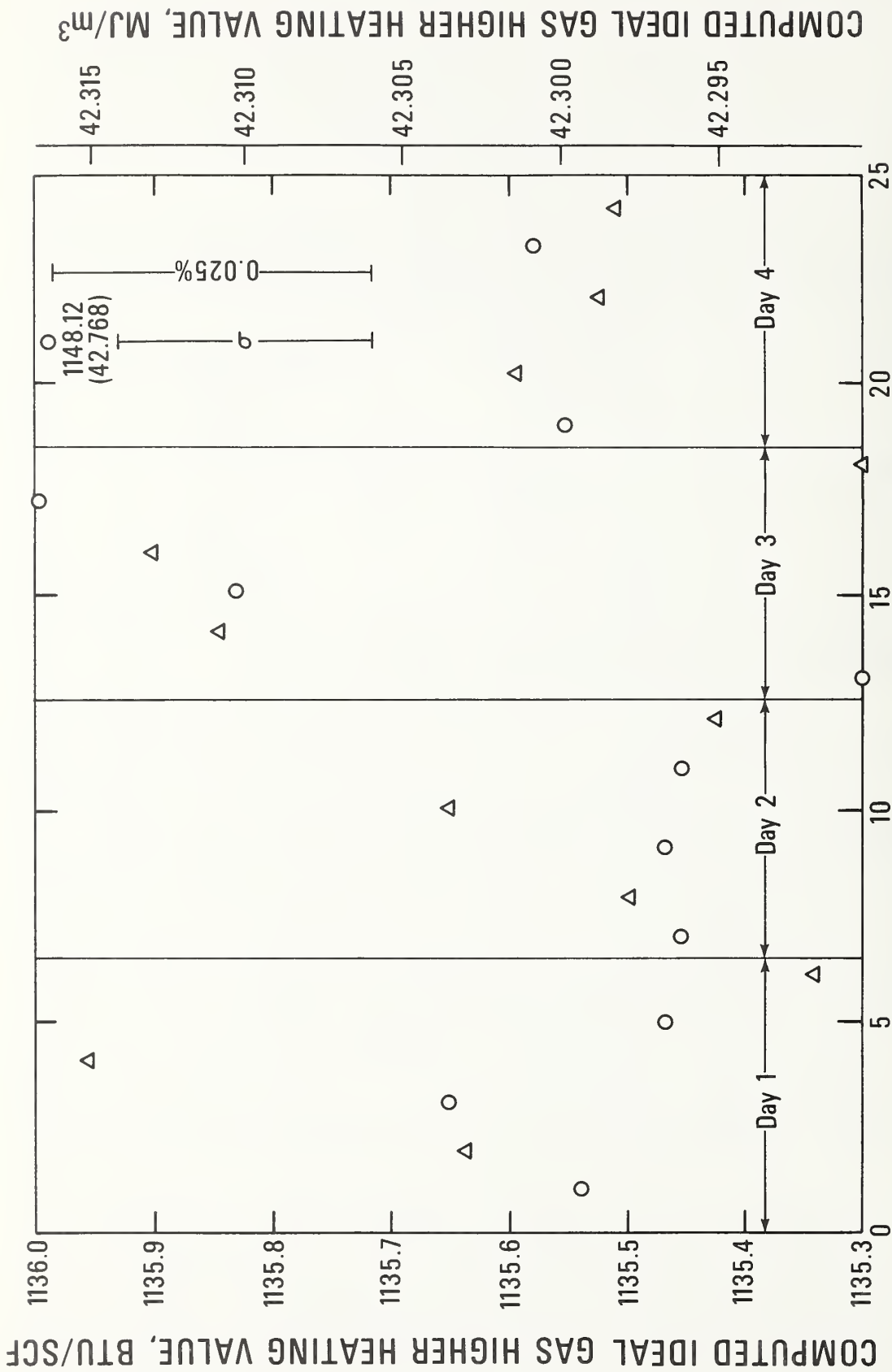


Figure 4.3. Computed ideal gas heating values for mixture E. The symbols O and Δ denote operators 1 and 2, respectively. The trye heating value for this mixture is 1135.29 Btu/scf (42.2906 MJ/m³).

this time using only one operator and one gas mixture. Figure 4.4 shows the computed heating values for the 44 analyses which were taken over a two day period. This time the total spread of computed heating values was 0.07% and the largest deviation in heating values was 2.8 estimated standard deviation; 75% of the analyses were within one estimated standard deviation of the average. This indicated that the malfunctioning integrator had caused the outliers.

Table 4.5 lists the biases and estimated standard deviations obtained from the results of the above two tests; values differing from the average measured value by more than four estimated standard deviations were omitted in the computations. Comparing the biases of mixture G with those of the other mixtures points out the necessity of using a calibration gas which is similar to that of the unknown mixture. Also, the estimated standard deviations were generally larger for mixture G; this indicates that the operating variables would require some modification when analyzing mixtures containing large amounts of nitrogen and/or ethane.

Figure 4.3 and 4.4 illustrate how an insufficient number of calibration runs can introduce biases into the computed heating values. Since the calibration gas was the analyzed gas in these figures, the biases had to be caused by the calibration. In the first test all results were based on a single calibration run at the beginning of the day while two calibrations were made at the beginning of each day in the second test. We recomputed the biases for the second test by using the first three runs of each day for calibration; the bias dropped from 0.009 to 0.005 percent. Thus, it appears that at least three good (i.e., not a set that includes an outlier) calibration runs should be used to minimize biases introduced by calibration. This assumes that the other parameters in the GC system remain constant. Since this may not always be the case, calibrations should be made several times during the day as an additional check.

4.3. Evaluation of Gas Chromatograph Column Configuration and Digital Integrators

The previous section determined the precision and accuracy of one gas chromatograph (GC)-integrator system. Additional tests employing different column configurations and integrators were needed to determine the factors limiting the precision of GC analyses. This section describes tests using two different three column configurations and two different GC's, and tests directly comparing two different integrators. Table 4.6 summarizes the chromatograph-integrator combinations used in all of the analysis tests.

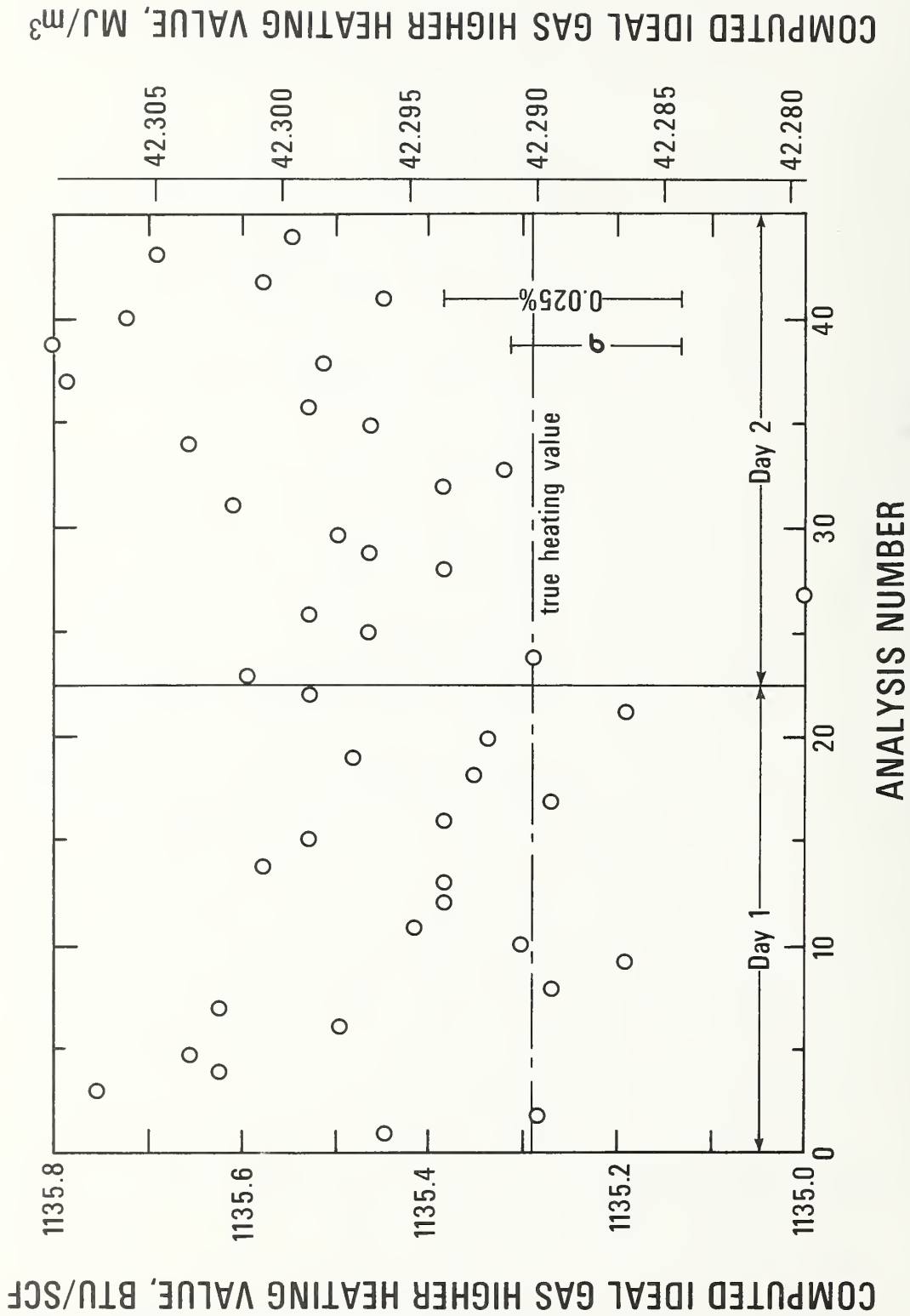


Figure 4.4. Computed ideal gas heating values for mixture E after integrator repair.

Table 4.5. Estimated bias and standard deviation of the composition and ideal gas heating value for four mixtures analyzed by gas chromatography.

Mixture	Number of Analyses	Estimated Bias ¹ and Standard Deviation (s) in														Heating Value Btu/scf (MJ/m ³)			
		Composition, mol %																	
		N ₂		CH ₄		C ₂ H ₆		C ₃ H ₈		iC ₄ H ₁₀		nC ₄							
		B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s		
C	24 ³	0.0133 + 0.0007	0.0011	-0.0449 + 0.009	0.014	0.016 + 0.064	0.010	0.004 + 0.004	0.007	0.004 + 0.001	0.003	0.003 + 0.002	0.004	0.10 + 0.10 + 0.004 + 0.004	0.16 (0.006)				
E ²	24 ³	0.0006 + 0.0009	0.0013	-0.023 + 0.0098	0.016	0.012 + 0.008	0.012	0.003 + 0.004	0.006	0.004 + 0.002	0.004	0.005 + 0.004	0.006	0.31 + 0.14 + 0.012 + 0.005	0.22 (0.008)				
F	24 ³	-0.0023 + 0.0010	0.0014	-0.025 + 0.006	0.010	0.015 + 0.004	0.006	0.009 + 0.004	0.007	0.004 + 0.002	0.003	0.005 + 0.003	0.005	0.46 + 0.13 + 0.017 + 0.005	0.22 (0.008)				
G	24 ³	-0.114 + 0.013	0.006	0.131 + 0.031	0.044	-0.003 + 0.051	0.034	0.011 + 0.010	0.017	0.008 + 0.004	0.003	0.004 + 0.003	0.005	1.27 + 0.28 + 0.047 + 0.010	0.39 (0.015)				
E ²	44 ⁴	-0.0004 + 0.0012	0.0019	-0.011 + 0.009	0.017	0.006 + 0.006	0.012	0.000 + 0.002	0.006	0.003 + 0.001	0.002	0.002 + 0.001	0.003	0.10 + 0.07 + 0.007 + 0.003	0.17 (0.006)				

¹Biases are given with 99% confidence interval.

²This mixture was used as the calibration gas.

³Analyses taken prior to integrator repair and on four different days; outlying points were omitted from computation of bias and estimated standard deviation.

⁴Analyses taken after integrator repair and on two different days.

Table 4.6. Summary of gas chromatograph-integrator systems used in analysis tests.

	Two Column Configuration	Three Column Configuration	
		Configuration A	Configuration B
Chromatograph	I	II	I
Detector	Hot-wire	Thermistor	Hot-wire
Automatic Valve Switching	No	Yes	No
Integrator	A	A & B	A

4.3.1. Selection of Column Configurations and Column Packings

Obtaining the most accurate and precise analysis of vaporized LNG samples requires:

- a) resolving oxygen, nitrogen and methane through pentane and column back flushing (i.e., carrier gas flow reversal) to obtain a composite C_6+ peak,
- b) analyzing for all components in a single sample injection,
- c) completing the analysis within approximately 30 minutes,
- d) operating the column ovens in an isothermal mode,
- f) minimizing the effect of baseline upset by valve switches, and
- g) computing the composition by using peak areas.

The primary purpose in resolving the nitrogen and oxygen peaks is to detect contaminated samples since the most likely contaminant is air. Also, it is a good way to detect leaks in the GC plumbing. The need for good resolution between the hydrocarbons is obvious. The anticipated concentration of hexane and heavier components is small (< 0.01 mol percent); therefore, combining the C_6+ fraction into one peak by backflushing causes an insignificant error in the computed heating value.

The GPA [17] and ASTM [6] analysis procedures suggest using two sample injections -- one injection into a column to resolve air and methane through pentane followed by a backflush for the C_6+ peak; a second injection into a different column to resolve nitrogen, oxygen and methane (the column then is backflushed to remove the remaining hydrocarbons). However, the two analyses must be combined. This is done through sample size or methane peak area. Both methods diminish analytical precision.

The primary reason for minimizing the analysis time is that the peak height to peak width ratio decreases with increasing residence time. Maximum accuracy and precision in peak area measurement decreases as the ratio decreases. It was found that, for the column configurations and operating parameters considered here, the analysis times fell in two ranges -- one was 40 minutes or greater and the other was 24 to 30 minutes. Configurations

having long analysis times elute the C_6+ peak last; the other configurations elute methane last.

Temperature programming is an attractive way of reducing analysis time and sharpening peaks of the heavier components. However, whenever feasible, isothermal column oven operation is normally preferred over temperature programming when using thermal conductivity detectors. This is because flow variations caused by changing column temperature produce baseline drift when using these detectors. Temperature programming becomes attractive if the baseline drift can be compensated for reliably by the integrator or if flame ionization detectors are used. These detectors are insensitive to flow variations but also do not detect nitrogen, oxygen and carbon dioxide.

Multicolumn configurations require valve switching with its associated carrier gas flow upsets; these, in turn, produce baseline upsets. The time duration of baseline upsets are minimized by:

- a) adjusting flow restrictors in column bypass lines so that the steady-state carrier gas flow rate remains constant after switching the column in or out, and
- b) using carrier gas inlet pressure instead of mass flow to control the flow rate [18].

To minimize peak integration problems, each valve switch should occur far enough away from the nearest peaks to allow baseline establishment between the peaks and the valve switch.

Traditionally, peak height measurement was preferred over peak area measurement for computing composition from chromatograms. Peak height was used because it was easy, quick and usually more precise than peak area. However, the peak height response is linear over a narrow concentration range, using a 0.5 cm^3 sample loop volume, peak height response is linear at concentrations below roughly 10 mol percent (peak area response is close to linear over the entire composition range). Also, peak heights cannot be used with composite backflush peaks. The currently available programmable integrators eliminate the drawbacks to using peak areas. Also, since both methane and C_6+ peaks must be measured, peak area measurement was selected.

Compromises are necessary to meet the requirements listed above. It is possible to make the complete analysis on one column [19], but it requires temperature programming between -70°C and 145°C and the C_6+ backflush peak retention time is about 22 minutes; the C_6+ peak width is roughly 3 minutes which, in combination with the baseline drift caused by temperature programming, can cause errors in the peak area measurement. If the separation of oxygen and nitrogen is not required, the analysis can be made by temperature programming between 35 and 145°C . In this case the analysis time is roughly 15 minutes. Based on the data for five replicate natural gas analyses [19], the estimated standard deviation in the computed heating value for this method is 0.04 percent.

The most commonly used column packing for isothermally resolving nitrogen and oxygen is molecular sieve 5A [18]. It makes a good separation between nitrogen and methane, but cannot be used with the higher hydrocarbons. This means that at least two columns in series are needed -- one to separate oxygen, nitrogen and methane (molecular sieve) and one to resolve the higher hydrocarbons (we used DC 200/500 on Chromasorb PAW). Using this configuration without temperature programming, the retention time for the pentanes is roughly 20 minutes. After eluting the pentanes, the DC 200/500 column is backflushed with the C₆+ peak eluting in another 20 minutes; this peak becomes so broad that at the 0.01 mol percent level it would not be detected using a TC detector and a 0.5 cm³ sample loop.

When operating the columns isothermally the preferred way to shorten the pentane and C₆+ retention times is to use two columns to resolve the C₂+ hydrocarbons. By selecting the proper column packings, lengths, and temperatures, and by using column bypassing, the components can be eluted in almost any sequence desired. In this work we considered two three-column configurations -- one which eluted the pentanes and C₆+ peaks midway through the analysis (configuration A) and one which eluted them at the beginning of the analysis (configuration B).

4.3.1.1. Column configuration A

Figure 4.5 shows a schematic of this three column arrangement and figure 4.6 is a chromatogram of a sample taken during a flow facility sampling test; table 4.7 lists the operating parameters for this configuration. The vaporized LNG samples were analyzed using the following automatic valve switching sequence:

- Step 1. Switch valve V1 for sample injection -- all columns are in series.
- Step 2. Switch valve V2 to bypass columns 2 and 3 after oxygen, nitrogen and methane have entered these columns; ethane through pentanes are eluted in this step. (Restrictor A is adjusted so that its pressure drop is comparable to the pressure drop of columns 2 and 3; this minimizes baseline upset after valve switchings.)
- Step 3. Switch valve V1 to original position to start backflush.
- Step 4. Switch valve V2 to put columns 2 and 3 back in series with column 1.
- Step 5. Switch valve V3 to bypass column 3 which now contains oxygen, nitrogen and methane; ethane (and carbon dioxide, if present) are eluted in this step. (Restrictor B is set to provide the same pressure drop as column 3.)
- Step 6. Switch valve V2 to bypass column 2; the C₆+ peak elutes in this step.

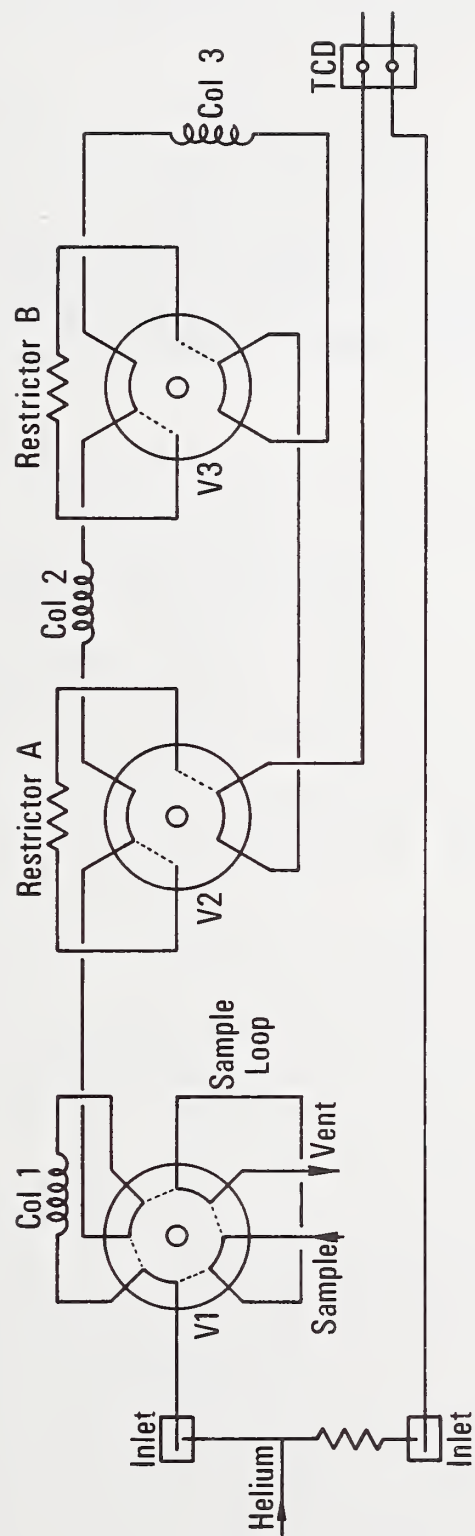


Figure 4.5. Schematic of three column configuration A.

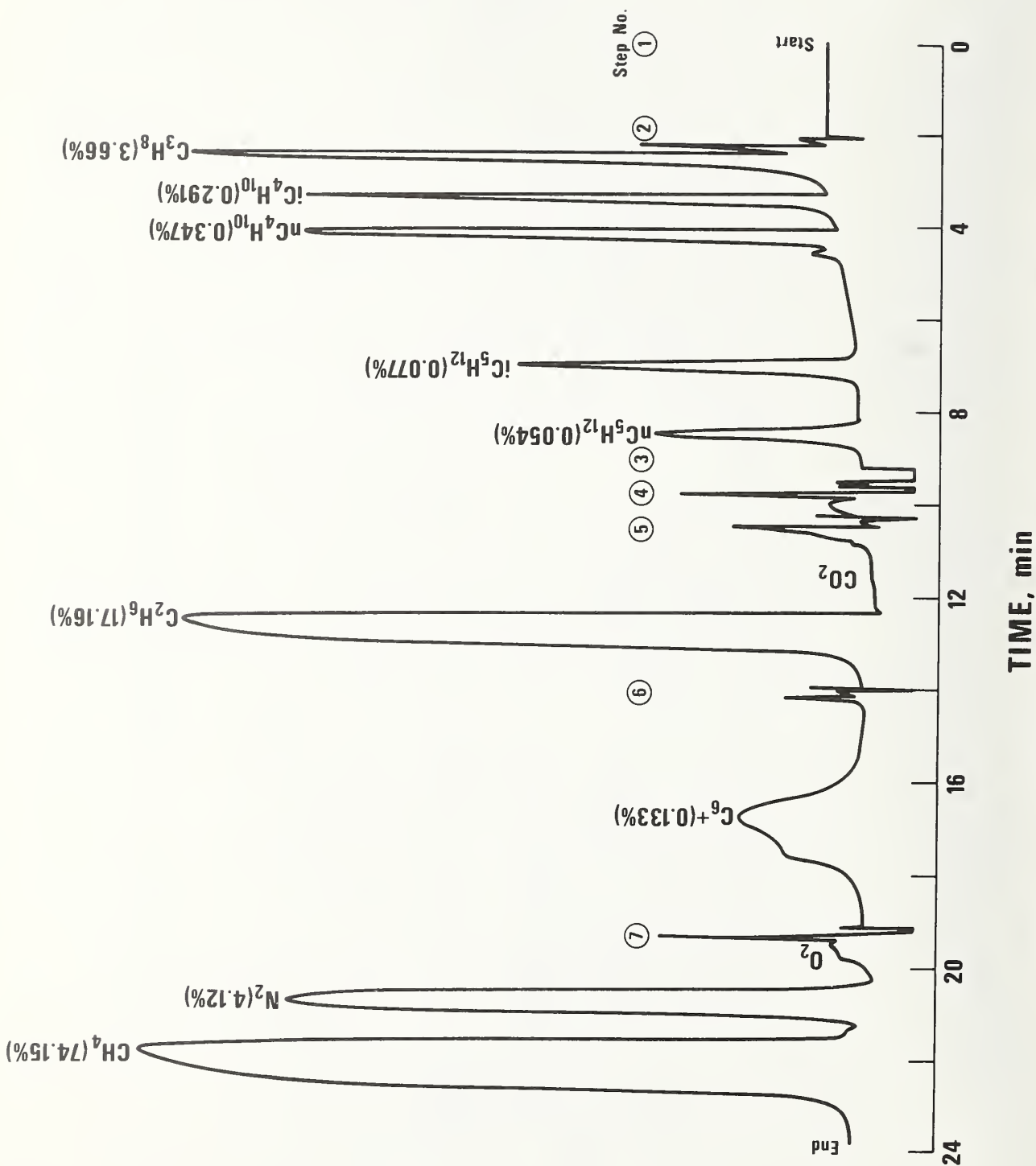


Figure 4.6. A chromatogram of a natural gas mixture using column configuration A. The peaks are shown on a logarithmic scale.

Step 7. Switch valves V2 and V3 to put column 2 and 3 in series; oxygen, nitrogen and methane elute in this step.

Table 4.7. Operating conditions for Column Configuration A.

Carrier gas flow rate	27.5 scm ³ /min
Sample loop volume	0.5 cm ³
Column 1 temperature (10 ft. (3 m) x 1/8 in. (3.2 mm) of 15% Squalane on Chromosorb PAW, 60/80 mesh)	66°C
Column 2 temperature (5 ft. (1.5 m) x 1/8 in. (3.2 mm) of Porapak N, 60/80 mesh)	66°C
Column 3 temperature 5 ft. (1.5 m) x 1/8 in. (3.2 mm) of Molecular sieve 5A, 60/80 mesh)	66°C
Detector temperature	66°C
Detector current (Thermistor detector)	

Table 4.8 lists the results of a series of analyses made using this configuration and prepared mixture H (see table 4.2). A comparison of these results with results given in table 4.4 shows that the precision of the two different column configurations was comparable. No biases were found in this test since the same mixture was used for both calibration and analysis; three calibrations were made prior to running the replicate samples. It is encouraging that configuration A, with eight valve switches, had about the same precision as the much simpler two column system which had only three valve switches.

Unfortunately, the backflush peak in configuration A was so broad that one of the two integrators (integrator A) had difficulties in accurately measuring the C₆+ peak area at the 0.10 mol percent level (see sections 5.2.3.1). Although integrator B reliably integrated the C₆+ peak, the broad backflush peak affected the reliability of integrating the following two peaks. It is extremely unlikely that the integrators would reliably detect a C₆+ peak at the 0.01 mol percent level. (Missing a C₆+ concentration of 0.01 mol percent produces a 0.04 percent error in the computed heating value.)

The only way to obtain a relatively precise C₆+ analysis is to reduce the retention time; this can be done by choosing a different three-column configuration.

4.3.1.2. Column configuration B

If precise determination of pentanes and C₆+ concentrations in the 0.01 mol percent range is required, these components must be eluted at the beginning of the analysis. This is done by using the columns listed in table 4.9 and the flow scheme shown in figure 4.5. Configuration B uses the following valve sequence:

Table 4.8. Estimated standard deviation in composition and ideal gas heating value for prepared mixture H using two column configurations and two integrators.

Column Configur- ation	Integrator	Number of Points	Estimated Standard Deviation in									Heating Value %
			Composition, mol %									
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂		
A	A	12	0.007	0.006	0.003	0.005	0.0006	0.001	0.0008	0.0006	0.014	
	B	12	0.010	0.016	0.008	0.003	0.001	0.002	0.002	0.002	0.014	
B	A	7	0.007	0.020	0.017	0.007	0.0007	0.0008	0.0003	0.0008	0.06	

- Step 1. Switch valve V1 for sample injection -- all columns are in series.
- Step 2. Switch valve V2 to bypass columns 2 and 3 after all components except C₅+ have entered these columns; the iso and normal pentane elute in this step.
- Step 3. Switch valve V1 back to original position to backflush the C₆+ component.
- Step 4. Switch valve V2 to put columns 2 and 3 into series again.
- Step 5. Switch valve V3 to isolate oxygen, nitrogen and methane in column 3. Ethane through the butanes are eluted in this step.
- Step 6. Switch valve V3 to elute oxygen, nitrogen and methane.

Table 4.9. Operating conditions for Column Configuration B.

Carrier gas flow rate	28 scm ³ /min
Sample loop volume	0.5 cm ³
Column 1 temperature (6 ft. (1.8 m) x 1/8 in. (3.2 mm) of DC 200/500 on Chromasorb PAW, 60/80 mesh)	62 to 68°C
Column 2 temperature (25 ft. (7.6 m) x 1/8 in. (3.2 mm) of DC 200/500 on Chromasorb PAW, 60/80 mesh)	60°C
Column 3 temperature (6 ft. (1/8 m) x 1/8 in. (3.2 mm) of Molecular sieve, 5A, 60/80 mesh)	65°C
Detector temperature	120°C
Detector current	220 ma

Table 4.8 gives the results of a feasibility test for column configuration B; figure 4.7 is a chromatogram of a mixture containing a C₆+ fraction. The numbers indicate that this configuration produces more scatter than configuration A. However, these results are inconclusive because during these tests:

- a) there were temperature fluctuations in the column 1 oven (this oven was a valve oven and was not designed for precise temperature control);
- b) there were small air leaks which caused baseline shifts during the n-pentane and ethane peaks, (the shifts were found by running an analysis of the carrier gas) and
- c) there were indications that column 3 had been contaminated and needed to be regenerated; this caused extreme tailing of the methane peak.

Either of the latter two problems could produce the observed scatter in the computed heating value. Additional tests should be made to determine the precision of this configuration under better operating conditions.

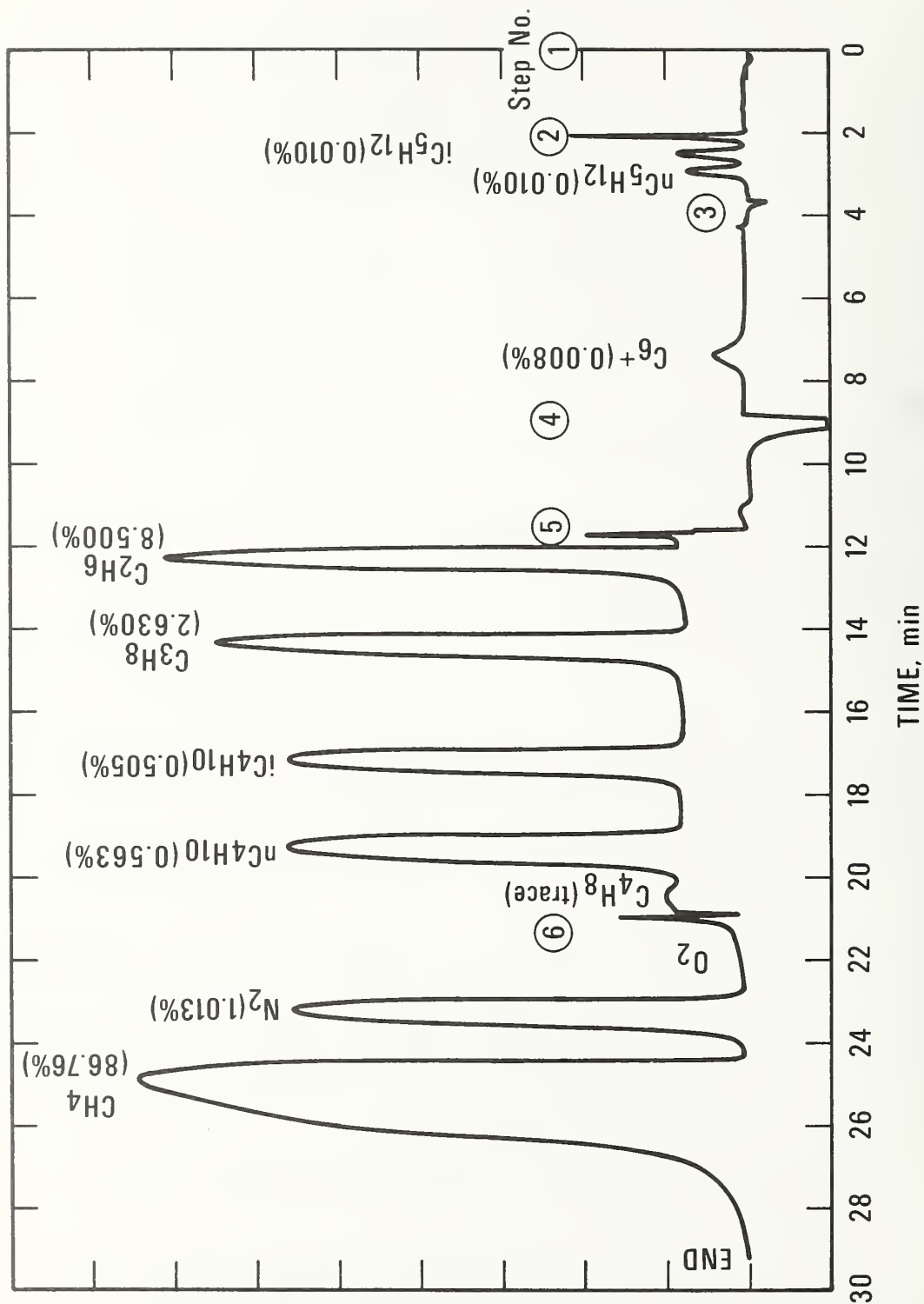


Figure 4.7. A chromatogram of a natural gas mixture using column configuration B. The peak heights are shown on a logarithmic scale.

Comparison of pentane peak heights showed that the peak heights using configuration B were twice that of configuration A. A sample containing C_6^+ was analyzed using configuration B; the C_6^+ peak eluted as a single sharp peak instead of the broad double peak produced by configuration A.

4.3.2. Digital Integrator Evaluation

The purpose of this work was to directly compare the accuracy and precision of two different programmable digital integrators. All digital GC integrators use voltage to frequency converters and counters to convert the detector output voltage into peak areas. The primary difference in integrators is in how they treat the converted signal. Both integrators used in this evaluation employed sophisticated software programs; these programs identified peaks, baseline segments and small peaks riding on the tail of larger peaks. Also, they compensated for transient baselines when computing peak areas. Both integrators were programable so that automatic adjustments, which were unique to the particular analysis, could be made during the analysis. The primary differences between the two integrators were that the software programs used different peak detection algorithms and that integrator B had considerably more programming capabilities; however, this versatility was unnecessary for analyzing vaporized LNG samples.

Table 4.8 shows the results of a direct comparison between the two integrators. For these tests, both integrators were connected directly to the GC detector output. Although integrator B has significantly greater scatter for some of the components, the precision in the heating value for both instruments was comparable.

During the last two days of a four day test period both integrators produced one or more extraneous results. The outliers appeared to occur randomly among the integrators which excluded the GC as the cause. We suspect that the outliers were caused by power fluctuations since the integrators were on different circuits; the GC detector power supply was connected to a constant voltage transformer. Shortly after these tests there were several power fluctuations severe enough to shut down the integrators.

4.4. Determination of the Effect of Operating Variables on Gas Chromatograph Analysis Accuracy and Precision

Variation in any of the operating parameters listed in table 4.7 could affect analysis precision and accuracy. However, variables such as column temperature will be electronically controlled. Therefore, they will be constant unless the set point is changed. The variables most subject to change are sample amount (i.e., sample pressure since a fixed volume sample loop is used) and carrier gas flow rate because it must be set manually.

Table 4.10 lists the results of two tests using column configuration A and both integrators. In one test we injected samples at pressures of 13.7, 21.1 and 8.6 psia (94,145 and 59 kPa); the calibration sample pressure was 13.7 psia (94 kPa). The table shows that the precision was insensitive to sample pressure. However, there was an apparent linear relationship between bias and sample pressure. This test indicates that maintaining the sample pressure within ± 10 percent of that used for calibration will keep the bias to less than 0.025 percent in the computed heating value.

The observed bias was caused by the method the integrators used to determine the peak area. Unless the peak was very large and sharp, part of the peak area was lost during the baseline determination. The absolute amount of area lost remained nearly constant as the peak area changed, provided that the peak width at the half-height did not change. Therefore, the relative amount of area lost decreased with increasing peak size. Since the methane peak was so large there was a negligible amount of area lost during baseline determination. However, this was not the case for all other components except the pentanes. (The pentane peak areas were so small that the effect was within the data scatter.) This explains why the apparent methane content decreased and the ethane through butane contents increased with increasing sample injection pressure.

In the second test the carrier gas flow was varied by ± 7 percent. Integrator A results were insensitive to the flow variation, whereas, integrator B showed a bias in the computed heating value. This bias was caused by the method used to establish baseline between the nitrogen and methane peaks. During these tests integrator B was programmed to force a baseline at a specified time. The time was set at the bottom of the valley between the two peaks (see figure 4.6) when the carrier gas flow rate was 26.5 scm³/min. Increasing the carrier gas flow rate made the valley bottom occur before the forced baseline. Therefore, the forced baseline caused the integrator to ignore part of the methane peak; this lowered the apparent methane content and raised the apparent composition of the other components. This problem can be eliminated by having the integrator automatically select the valley bottom as the baseline when two peaks are not completely resolved.

Based on these tests it appears that the precision and accuracy of the computed heating values are unaffected by changes of ± 7 percent in the carrier gas flow rate. This conclusion strictly applies only to configuration A. However, it should apply to other column configurations which have comparable time spacings between valve switches and peak elutions.

Table 4.10. Effect of varying sample pressure and carrier gas flow rate on the accuracy and precision of gas analysis;
column configuration A and prepared mixture H were used in this test.

Carrier Gas Flow Rate scm ³ /min	Sample Pressure kPa	Inte- grator	Number of Points	Estimated Bias ¹ (B) and Standard Deviation (s) in Composition, mol %																								Heating Value %																							
				N ₂												CH ₄														C ₂ H ₆				C ₃ H ₈				iC ₄ H ₁₀				nC ₄ H ₁₀				iC ₅ H ₁₂				nC ₅ H ₁₂	
				N ₂						CH ₄						C ₂ H ₆				C ₃ H ₈				iC ₄ H ₁₀				nC ₄ H ₁₀				iC ₅ H ₁₂				nC ₅ H ₁₂															
				B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s	B	s																
26.5 ²	94 ²	A	6	--	0.002	--	0.003	--	0.004	--	0.005	--	0.005	--	0.005	--	0.005	--	0.005	--	0.005	--	0.005	--	0.005	--	0.005	--	0.005	--	0.005	--	0.009	--	0.006	0.006															
		B	6	--	0.006	--	0.005	--	0.008	--	0.003	--	0.003	--	0.003	--	0.003	--	0.002	--	0.002	--	0.002	--	0.002	--	0.002	--	0.001	--	0.001	--	0.001	0.012																	
25.7	145	A	5	0.007 +0.003	0.001	-0.128 +0.019	0.008	0.084 +0.005	0.002	0.027 +0.010	0.008	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.004 +0.0003	0.004	0.01																	
		B	5	--	0.002	-0.125 +0.011	0.048	0.087 +0.011	0.005	0.024 +0.005	0.002	0.002 +0.005	0.003	0.003 +0.002	0.003	0.003 +0.002	0.003	0.003 +0.002	0.003	0.003 +0.002	0.003	0.003 +0.002	0.003	0.003 +0.002	0.003	0.003 +0.002	0.003	0.003 +0.002	0.003	0.003 +0.002	0.003	0.003 +0.002	0.001	0.007																	
26.7	59	A	5	--	0.005	0.093 +0.010	0.008	-0.065 +0.011	0.005	-0.016 +0.012	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	-0.011 +0.011	0.005	0.007																	
		B	5	--	0.012	0.072 +0.035	0.015	-0.055 +0.011	0.005	-0.023 +0.007	0.003	-0.023 +0.007	0.003	-0.023 +0.007	0.003	-0.023 +0.007	0.003	-0.023 +0.007	0.003	-0.023 +0.007	0.003	-0.023 +0.007	0.003	-0.023 +0.007	0.003	-0.023 +0.007	0.003	-0.023 +0.007	0.003	-0.023 +0.007	0.002	-0.09 +0.02	0.01																		
26.5 ²	145 ²	A	5	--	0.001	--	0.006	--	0.003	--	0.006	--	0.003	--	0.006	--	0.006	--	0.006	--	0.006	--	0.006	--	0.006	--	0.006	--	0.006	--	0.006	--	0.004	0.01																	
		B	5	--	0.004	--	0.006	--	0.004	--	0.002	--	0.002	--	0.002	--	0.002	--	0.002	--	0.002	--	0.002	--	0.002	--	0.002	--	0.002	--	0.008	0.009																			
24.2	145	A	5	0.010 +0.005	0.002	-0.022 +0.005	0.010	-0.022 +0.005	0.007	0.021 +0.009	0.004	0.001 +0.001	0.006	0.001 +0.001	0.006	0.001 +0.001	0.006	0.001 +0.001	0.006	0.001 +0.001	0.006	0.001 +0.001	0.006	0.001 +0.001	0.006	0.001 +0.001	0.006	0.001 +0.001	0.006	0.001 +0.001	0.006	0.001 +0.001	0.008	0.01																	
		B	5	--	0.006	0.065 +0.035	0.015	0.065 +0.035	0.008	--	0.005	--	0.008	--	0.008	--	0.008	--	0.008	--	0.008	--	0.008	--	0.008	--	0.008	--	0.008	--	0.008	0.07																			
27.6	145	A	5	0.008 +0.006	0.002	--	0.027	--	0.009	--	0.023	--	0.009	--	0.023	--	0.023	--	0.023	--	0.023	--	0.023	--	0.023	--	0.023	--	0.023	--	0.023	--	0.004	0.03																	
		B	5	0.016 +0.006	0.002	-0.165 +0.035	0.024	0.105 +0.035	0.015	0.032 +0.014	0.006	0.105 +0.035	0.015	0.032 +0.014	0.006	0.105 +0.035	0.015	0.032 +0.014	0.006	0.105 +0.035	0.015	0.032 +0.014	0.006	0.105 +0.035	0.015	0.032 +0.014	0.006	0.105 +0.035	0.015	0.032 +0.014	0.001	0.12 +0.05																			

¹Biases are given with 99% confidence interval; dashed line means that the confidence interval was larger than the bias.

²GC was calibrated at these conditions prior to making this and the two sets of analyses.

4.5. Sample Handling Procedures

Typically, the LNG sampling procedure will include the filling of gas sample containers. Therefore, we need to know which filling procedures are the most reliable and if there are any effects of storage which could change the sample composition over a period of time. Considering the filling procedures described by Miller [12] (see discussion in Section 3.2) and our own past experiences, we feel that the fill and purge technique along with purging at a constant flow rate are the simplest and most reliable; however, when using the latter technique, some method of flow measurement is desirable.

In using the purge and fill technique we must know how many purges are adequate to remove the original gas. Miller [12] states that 3 to 8 are sufficient; the ASTM standard [13] recommends 10 at one point and 3 to 8 in another. The number depends upon the line pressure because the important factor is the number of sample container volumes of gas, at ambient pressure (assuming ideal gas behavior), which pass through the sample container. Assuming complete mixing, 10 volumes of gas should be adequate. However, we felt that a test was necessary to determine:

- a) the number of purges required, both in filling the sample cylinder and in purging the lines to the GC, and
- b) the effect of sample "aging" which could change the apparent composition.

4.5.1. Test Apparatus and Procedure

Figure 4.8 shows a schematic of the apparatus for evaluating sample handling techniques. In the first test we determined the number of purges necessary to reduce the air concentration to below the detectable limit on the GC. We used 150 cm³ stainless steel sample cylinders with valves at each end; the valves contained teflon packing and Kel-F stem tips. The valves were fitted with a gland and nut for making an O-ring coupling to the sample line. The sample cylinders were not pretreated in any way. However, they were pressure checked to 1500 psi (10 MPa) and vacuum checked with a helium leak detector. In this test, we attached a sample cylinder to valve V5. Initially, several analyses were made directly from the prepared mixture cylinder. In addition to noting the nitrogen content of the mixture, we also recorded the ratio of the methane to nitrogen peak areas. (This is a sensitive method for detecting air leaks.) To make sure that air was in the sample cylinder initially, we evacuated the manifolding between valves V2 and V3, with V5 and V6 open, but V7 closed; after closing V5, V7 was opened to let air in. Next the manifolding was pressurized to 30 psig (0.3 MPa) with the mixture by opening valves V1 and V2. To purge and fill the sample cylinder we would alternately open valve V5 with V7 closed, then close V5 and open V7; V7 was left open just long enough to bleed the pressure

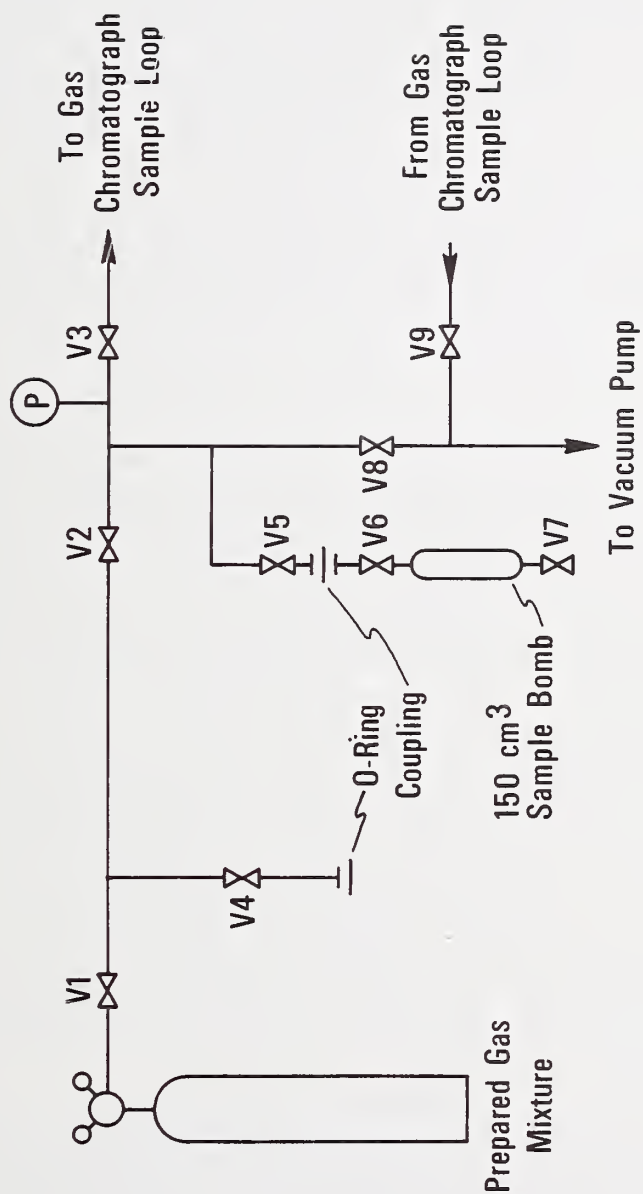


Figure 4.8. Flow schematic for sample handling tests.

to near ambient. After the desired number of purges, valve V2 was shut and the manifold evacuated through V8. During this evacuation, valve V5 was open but V6 closed. Next we cracked open valve V6 to fill the manifold to 2 psig (0.11 MPa). After filling the manifold, valve V5 was closed and V6 opened. Opening valves V3 and V9 evacuated the manifold and GC sample loop. After V9 was closed we cracked open valve V5 to fill the line with sample gas and the analysis started. Originally the GC sample loop was purged several times, but tests showed that one 20 to 30 second evacuation and fill was sufficient. However, if there was sufficient pressure in the sample cylinder, we repeated the evacuation and fill at least once.

4.5.2. Evaluation of Results

Analyses were taken on samples from cylinders purged and filled 4, 6, 10 and 14 times. Only the last one indicated that all of the air was removed. This is equivalent to 42 sample container volumes of gas at ambient conditions, four times more than would be expected if there was complete mixing.

In the second test we looked at sample aging to see if processes such as adsorption on sample cylinder walls would alter sample composition. Again we used sample cylinders initially containing air and used 14 purge and fills with a sample pressure of 30 psig (0.3 MPa). However, this time the sample containers were filled at the O-ring coupling adjacent to valve V4; for analysis we attached the sample cylinders to valve V5 and followed the line and GC sample loop purging procedure outlined above. To ensure that the change in composition, if any, was not due to biases in the GC analysis, the prepared mixture from the cylinder was analyzed before and after each sample cylinder analysis. Analyses were made at 1/2, 4, 24, 48 hours and 50 days after filling; in all cases the samples were within one estimated standard deviation of the original mixture for all components.

Additional tests (see section 5.2.1.1) showed that continuous purging also was a satisfactory sample collecting technique. However, the sample container was purged the equivalent of 14 or more purge and fills. The gas throughput was estimated by monitoring the pressure, flow rate (5 to 11 standard liters per minute) and time.

4.6. Conclusions and Recommendations

4.6.1. Accuracy and Precision of Analysis by Gas Chromatography

Based on the results of comparative tests it was found that a gas chromatograph-digital integrator system was more accurate and precise than mass spectrometry. Additional tests showed that the gas chromatograph produced a bias (i.e., the total uncertainty consists of only analytical precision) of 0.02 percent, or less, in the computed heat value provided that:

a) the response factors for computing the composition are the average of three or more calibrations,

b) the gas chromatograph and digital integrator operating parameters remain constant during calibration and analysis runs, and

c) the calibration and unknown mixture compositions are comparable.

Test results showed that gas mixtures within the anticipated LNG composition range given in table 2.1 can be analyzed by gas chromatography using only one calibration gas (which is within the same range) without producing biased results.

The total uncertainty in computing the heating value routinely can be less than ± 0.1 percent. This assumes:

a) taking three or more replicate analyses and averaging the computed heating value,

b) making three or more calibration runs prior to the analyses and averaging the resulting response factors for each component,

c) using a calibration gas which has a total uncertainty in the composition which, neglecting the specific heating value of each component, corresponds to an accuracy of ± 0.03 percent in the computed heating value (if the calibration gas is prepared by a commercial vendor, an independent verification of the composition should be made),

d) using a gas chromatograph-integrator system with a known precision of 0.06 percent, or better (based on three standard deviations), in the computed heating value, and

e) following the sample injection procedures described previously (sections 4.1.1.2 and 4.1.1.3) for gas analysis.

Appendix 7.3 outlines how to compute the total uncertainty and how the number of calibration and analysis runs affect the uncertainty; also, it provides a method for rejecting a given analysis.

If precise analysis of C_5+ is unnecessary, column configuration A should be used for analyzing vaporized LNG samples. If the pentanes and C_6+ peak areas need to be precisely known, column configuration B is recommended; however, further tests are needed to insure that the configuration will be as precise as configuration A. Also, other column configurations need to be evaluated.

To maintain the maximum accuracy and precision, the sample amount and carrier gas flow rate should be kept constant to within ± 7 percent. It is recommended that a high quality rotameter be used to monitor the flow rate; the rotameter can be calibrated with a simple soap bubble flowmeter.

Direct comparison of two different integrators showed that the two instruments had comparable precision. Based on all of the tests (see section 4.3.1.1 and 4.3.2), we conclude that, at present, the digital integrator is less precise than the gas chromatograph employing a thermal conductivity detector. Therefore, the most precise analysis will be one in which the integrator "sees" the sharpest

peaks and the least baseline fluctuations before and after peaks. To maximize the precision, a programmable integrator should be used.

4.6.2. Sample Handling

Tests showed that both the purge and fill technique and the continuous purge technique were accurate methods for collecting vaporized LNG samples. However, both techniques require some care to insure that the cylinder has been purged properly.

Commercially available stainless steel sample cylinders are recommended for collecting gas samples. No special preparation of the cylinders is required prior to their use. Tests showed that stainless steel cylinders do not alter the hydrocarbon content over a six week period. (However, stainless steel is not inert to sulphur compounds.)

4.6.3. Recommended Criteria for Selecting a Gas Chromatograph-Integrator System

Based on our experience we recommend that the following features be included in any gas chromatograph-integrator system dedicated to routine analysis of vaporized LNG samples:

- a) Automatic valve switching - this is required to insure that the right valve is switched at the right time.
- b) Thermal conductivity detector -- this is the only detector that is sensitive to all the components in natural gas and is linear in response over the anticipated concentration ranges (i.e., the output signal is directly proportional to the concentration). Hot wire filaments are preferred over thermistors because they provide superior stability, longer operating life and fewer anomalies of operation [20]. The instrument should contain a thermal protect device to minimize the risk of oxidizing the filaments. To maximize sensitivity the detector cell volume should be as small as possible.
- c) Isolated power supply -- this is necessary to prevent electrical noise generated by oven temperature controllers from entering the detector circuitry. The detector power input should be connected to a constant voltage transformer to minimize effects of line power fluctuations.
- d) Carrier gas flow control by inlet pressure -- this is preferable to mass flow control when the analysis involves valve switching [18]. A two-stage pressure regulator should be used to keep the inlet pressure constant.
- e) Readily accessible valve and column connections -- this is needed to make routine maintenance and leak checking easy.
- f) Programmable digital integrator -- this is necessary to maximize the analytical precision. A memory protect feature should be used

to prevent losing programs during momentary power failures. A constant voltage transformer should be connected to the integrator to minimize noise due to line power fluctuations.

4.6.4. Recommendations for Future Work

If C_5 hydrocarbons must be analyzed precisely, additional testing of column configuration B is needed to determine the analytical precision of the method. Other column packings should be considered for column 1 of this configuration.

A survey of the open literature shows that there is no widely accepted method for analyzing vaporized LNG samples. An extensive study should be made to determine the best gas chromatographic technique for analyzing LNG type mixtures. The study should include selecting the best column packings, column configurations and detectors. Capillary columns should be considered because these columns produce extremely sharp peaks which are ideal for use with digital integrators. Once the gas chromatograph system has been selected, extensive tests should be made to establish representative values for the precision and accuracy of the technique.

5.0 LNG SAMPLING

The sampling tests included both laboratory and field tests. The laboratory tests provided close control of all operating variables whereas the field tests permitted evaluation of a full scale sampling system.

The chronological order of the tests and the variables considered for their effect on sampling accuracy and precision were:

- a) laboratory tests (Probe Evaluation) - sampling rate, LNG flow rate, temperature, pressure, degree of subcooling and three probes;
- b) laboratory tests (Vaporizer Evaluation) - sampling rate, temperature, degree of subcooling, LNG flow rate and two vaporizer designs,
- c) field tests (LNG Flow Facility Test 1) - three probes and three vaporizers,
- d) laboratory tests (General Tests) - accumulator residence time, vaporizer outlet temperature, pressure drop upstream of vaporizer, heat leak to sample probe and presence of heavy components (C_5 +) in the LNG,
- e) field tests (Shipboard Tests) - sampling rate and comparison between liquid and vapor sampling,
- f) field tests (LNG Flow Facility Test 2) - sampling rate, heat leak, start-up transients and three variations of a vaporizer design.

This section discusses all the laboratory tests before describing the field tests.

5.1. Laboratory Tests

5.1.1. Laboratory Facility

This section discusses all aspects of the laboratory facility with the exception of the gas analysis equipment; this is discussed in section 4.1.1.

5.1.1.1. Laboratory Scale Apparatus

Figure 5.1 shows a schematic of the laboratory scale apparatus used in this work. The cryogenic portion of the apparatus was contained in an evacuated double walled dewar which was 33 inches (840 mm) deep and had a 10-3/4 inch (270 mm) inside diameter. The LNG vessel, which was 8-1/2 inch (215 mm) long with an outer diameter of 8 inches (203 mm), had an inner volume of roughly 6 liters; the wall was copper and the two end plates were brass. A centrifugal pump circulated LNG through a turbine flowmeter, with a range of 0.25 to 2.5 gpm (0.016 to 0.16 L/s), and through the test section between valves T1 and T2. All of the lines in this loop were 1/2 inch (12.7 mm) O.D. copper tubing except for a 3 inch (76 mm) long section containing 1 inch (25 mm) O.D. copper tubing just downstream of valve T1. This short section acted as a mixing chamber to reduce flow turbulence caused by the valve. The test section was a 6-3/4 inch (171 mm) long straight section. It contained two probes -- an upstream facing pitot tube and a side tap. Both probes were mounted vertically and were made of 1/16 inch (1.59 mm) O.D. by 0.023 inch (0.58 mm) I.D. stainless steel tubing. There was roughly 6 inches (150 mm) of line between the probes and valves T4 and T5.

Besides the pitot tube and side tap, a "reference" probe was located in the straight-run leg of a tee positioned just upstream of valve T1. Since the flow into the tee was downward, the reference probe was in essence located on the bottom of the line. There were 8 inches (200 mm) of 0.023 inch (0.58 mm) I.D. stainless steel tubing between this probe and valve T3.

Valves T1 through T6 were bellows valves which were located inside the insulating vacuum space at LNG temperature. They were actuated by stem extensions protruding through the top plate of the apparatus. Valves T3 through T6 were mounted on a brass plate which was soldered to the top of the mixing chamber.

After a probe was selected by opening either valve T3, T4, or T5, the sample passed through 1/16 inch (1.59 mm) O.D. by 0.023 inch (0.58 mm) I.D. stainless steel tubing to one of three vaporizers. If the sample went to the reference vaporizer it first passed through valve T6 and 8 inches (203 mm) of tubing; valve T6 was a needle valve used to provide additional pressure drop to prevent sample backflash, i.e., flow reversal due to vaporization. The reference vaporizer consisted of a 5 inch (127 mm) long section of 1/16 inch (1.58 mm) O.D. by 0.051 inch (1.28 mm) I.D. stainless steel tubing and a 12 inch (304 mm) long section of 1/8 inch (3.18 mm) O.D. by 0.101 inch (2.56 mm) I.D. stainless steel tubing. The sample was vaporized

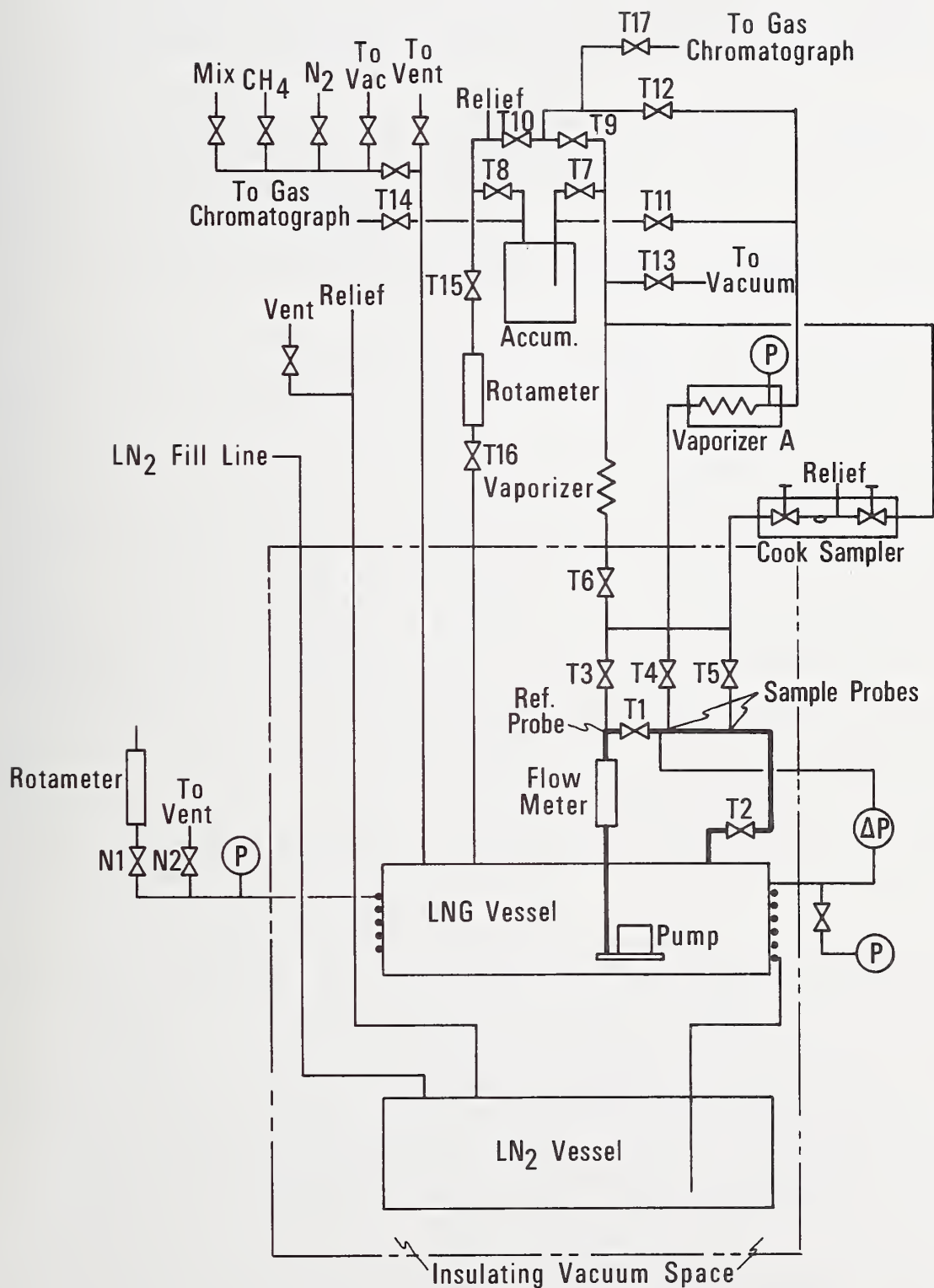


Figure 5.1. Schematic of laboratory scale apparatus.

by passing a low voltage, high amperage DC current through the two sections of tubing. To prevent shorts, the two ends of the vaporizer were epoxied into micarta couplings. The reference vaporizer was located less than 1/2 inch (13 mm) from where the sample line left the vacuum insulated space.

After the sample left the vaporizer it went to a manifold containing a 2 liter accumulator, through a 0 to 11.4 liter per minute (lpm) rotameter and back to the LNG vessel. The lines between the vaporizer and rotameter were 1/4 inch (6.35 mm) O.D. by 0.190 inch (4.83 mm) I.D. copper tubing; the copper tubing downstream of the rotameter was 3/8 inch (9.53) O.D. by 0.343 inch (8.71 mm) I.D. The accumulator could be isolated by closing valves T7 and T8; to maintain the sample flow, valves T9 and T10 (a regulating valve) were opened when the accumulator was isolated. Samples could be taken from the accumulator through valve T14 and from the bypass line through valve T17.

The second vaporizer, vaporizer A, contained an electrically heated vaporizing element, an adjustable back pressure regulator and a 0 to 30 psig (0.1 to 0.3 MPa) pressure gauge in a single unit. The heating element was a 1-1/4 inch (31.8 mm) wide by 28 inch (71.1 cm) long section of 100 mesh, 316 stainless steel screen wrapped around a 1/2 inch (12.7 mm) O.D. stainless steel tube. The diameter of the tube plus screen was 0.71 inches (18 mm). The vaporizer was designed to have the sample flow through the screen perpendicular to the axis of the tube. Also, the vaporizer contained a bypass valve and a pressure relief valve to protect the pressure gauge; both of these valves were capped in this study. Approximately 15 inches (381 mm) of stainless steel tubing connected the probes to vaporizer A. To prevent premature vaporization the line was vacuum jacketed to within 1 inch (25 mm) of the vaporizer inlet. When using this vaporizer, valves T11 and T12 replaced the function of valves T7 and T9.

The third vaporizer was a Cook sampler. Whereas the reference vaporizer and vaporizer A were for continuous sampling, the Cook sampler was designed for the batch sampling. Since the LNG sample was used to cool the sampler, the lines were piped to return the vapor to the LNG vessel; otherwise, the mixture would be depleted too quickly. The line from the probes to the sampler was roughly 15 inches (381 mm) long and vacuum jacketed.

Liquid nitrogen (LN_2) was used as the refrigerant in this study. The LN_2 vessel was 9-7/8 inches (251 mm) in diameter and 10-3/4 inches (273 mm) long and could contain about 10 liters of liquid. The LNG vessel was cooled by liquid flowing from the LN_2 vessel and through a coil wrapped and soldered on the LNG vessel. Cold nitrogen vapor leaving the coil went to another coil soldered on a 1/8 inch (3.2 mm) thick copper plate located roughly 2 inches (50 mm) below the top plate. All lines into the vacuum space were in thermal contact with this plate to reduce heat leakage. After

leaving the copper plate the nitrogen went to valve N1 and to a 0-44 liter per minute full scale rotameter and/or to bypass valve N2. When the apparatus was temporarily shutdown, e.g., overnight, nitrogen gas vented through another line which contained a relief valve set at 8 psig (0.16 MPa). During these periods the LN₂ vessel needed to be filled every 24 to 28 hours. The LNG vessel cooled at a rate of 1 to 2 K per day during idle periods.

To minimize radiation heat leak in the vacuum space, the apparatus was surrounded by a copper radiation shield. This shield was fastened to the LN₂ vessel and extended up to the copper plate.

5.1.1.2. Instrumentation

The temperature was measured using a platinum resistance thermometer (PRT) calibrated on the IPTS-68 temperature scale. The PRT was encased in a copper sheath pressurized with helium gas and positioned near the bottom of the LNG vessel. To measure the resistance we used a one ma constant current source with a potentiometer and a nanovolt null detector. The thermometer current was monitored by measuring the voltage drop across a 100 ohm standard resistor. Initially, we had the output from the null detector go to a power regulator; this regulator powered either a 120 or a 230 ohm heater wrapped on the LNG vessel. Although the temperature control was good (± 0.01 K), crosstalk developed between the power regulator and the null detector. This control method was dropped and thereafter the temperature was controlled by manually adjusting the LN₂ flow rate; using this method we could control the temperature to ± 0.03 K or better.

The saturation pressure of the LNG was measured with a 0 to 100 psia (0 to 6.9 MPa) quartz bourdon tube pressure gauge; this gauge was calibrated against an air dead weight gauge. The difference between the LNG pressure in the test section and in the LNG vessel was measured with a 0-300 inches of water (0 to 0.075 MPa) differential pressure gauge; this gauge was not calibrated during this project.

5.1.1.3. Safety features

Since the project involved flammable fluids all vents on the LNG system were tied to the room vent system. In addition, all vacuum pump outlets were connected to the same vent system. A relief valve, set at 120 psig (0.93 MPa), was located on the sample return line; this relief valve lead to the vent system. Finally, the insulating vacuum system had a relief valve connected to the vent and a pressure switch-solenoid valve combination. If the vacuum pressure increased to 100 microns (13 Pa), or, if there was a power failure the solenoid closed, isolating the vacuum space from the vacuum pump.

5.1.1.4. Recirculation pump motor

In the early stages of the project we had problems with motors to drive the recirculation pump. The same pump had been operated in liquid helium using a small three phase induction motor [21]. However, this motor had too much slippage when used with LNG (the density of LNG is four times greater than that of liquid helium). Two small motors in tandem still did not provide sufficient torque. Next we went to a 1/7 HP (106 watt) DC brush motor. The motor had sufficient torque but operated only 100 hours before the brushes failed. We replaced the brushes and used the same motor for another 90 hours. We then purchased and installed a larger three phase induction motor (1/2 hp (373 watt) at 10,000 rpm); it worked satisfactorily for the remainder of the project.

5.1.2. Experimental Procedure

5.1.2.1. Cooldown and Filling Procedure

After checking for leaks the insulating vacuum space was evacuated for 15 or more hours. By this time, the pressure was down to 10^{-4} mm Hg (0.1 Pa) or less. The complete LNG system was evacuated and back filled several times with high purity nitrogen which passed through a molecular sieve purifier. The cooldown was started by filling the LN_2 vessel with liquid nitrogen. With bypass valve N2 (see figure 5.1) wide open it took about 2 hours to bring the apparatus to 110 K.

Once at LNG temperature, all instruments and the pump were tested and the system was checked for cold leaks. If all was well the prepared mixture was condensed into the LNG vessel. Using a pressure of 40 to 50 psi (0.27 to 0.34 MPa) above the mixture's saturation pressure it took about one hour to fill the LNG vessel; when the LNG vessel was full there was a sudden rise in the pressure of the LNG system. At this point the startup was complete.

5.1.2.2. Preparation of Known Mixture

Because the accuracy of Sampling-Measurement Systems needed to be known, prepared mixtures of known composition were required in the laboratory scale apparatus. Of the several preparation methods considered, it was decided that the fastest and most accurate method was to fill a large (1.75 ft^3 (49.6 L)) gas cylinder with pure fluids and analyze the resulting mixture. (Size and weight limitations of the mass balance made preparation by weight unfeasible.) Partial pressures were used as a guide for estimating how much of each component should be added. Prior to filling, the cylinder was evacuated while being warmed with a heat lamp for at least 48 hours. The cylinder was filled to 1100 psi (7.59 MPa), which was well below the dew point pressure of the mixtures used here, and warmed with heat lamps for at least 6 hours. To enhance mixing, the cylinder was alternately heated in vertical and horizontal positions; when in the horizontal position a heat lamp was placed at each end of the cylinder. Next, a sample of the

mixture was analyzed on the GC; the cylinder then was heated for several more hours before making another analysis. If the analyses did not agree, the procedure was repeated until they did. (It never took more than 48 hours to obtain complete mixing.) Next, a series of at least 6 analyses were made on the mixture and the results averaged to determine the composition as accurately as possible.

After filling the LNG vessel the cylinder pressure was 100 to 150 psig (0.79 to 1.14 MPa). The mixture remaining in the cylinders was analyzed again to see if the composition had changed. Also, this gas mixture was analyzed several times during each operating day so that day to day shifts in the analysis could be detected. Table 5.1 gives the final sets of analyses for the prepared mixtures.

5.1.2.3. Operating procedure

The daily startup consisted of bringing the system to the desired operating condition and calibrating the gas chromatograph. The reference vaporizer and vaporizer A required 15 to 30 minutes, depending upon the sample flow rate, to establish steady-state conditions. The flow through the vaporizer had to be stable to obtain temperature control because the sample stream constituted 25 to 50 percent of the heat load on the system. (The only other major heat input was from the pump motor.)

After establishing the desired operating conditions the accumulator was isolated by closing valve T8 and valve T7 or T11 (see figure 5.1), depending upon the vaporizer used. (Operating the Cook sampler is discussed below.) To ensure a representative sample of the mixture leaving the vaporizer, the sample passed through the accumulator for at least 10 minutes under steady-state conditions. Immediately after isolating the accumulator either valve T9 or T12 was opened (valve T10 remained open) to avoid upsetting the system. At this time the operating variables could be changed so that the system would be stable when the next sample was ready to be taken. Under normal operation the power input to the reference vaporizer was set so that the tubing just downstream was warm to the touch; vaporizer A contained a thermostated heater set at 125°F (40°C).

To analyze the trapped sample in the accumulator the line between valve T14 and the GC was evacuated by opening valves V1, V5 and V6 (see figure 4.1) After closing valve V6, the line then was pressurized to about 2 psig (0.11 MPa) by opening valve T14. This purge and evacuate procedure was repeated three times; next valve T14 was left open and V1 closed. Then the analysis procedure described in section 4.1.1.3 was followed.

To draw a sample from the accumulator bypass line, valves V2 and V7 were opened to evacuate the line connecting V2 and T16. After closing V7, valve T17 was opened to pressurize the line to about 2 psig (0.11 MPa). After repeating the purge procedure three times, valve V2 was closed and T17

Table 5.1. Average value and estimated standard deviation in the composition, ideal gas heating value and liquid density of the prepared mixtures charged into the laboratory apparatus.*

Prepared Mixture Number	When Analyzed	Number of Analyses	Composition, mol %						Ideal Gas Heating Value Btu/scf (MJ/m ³)	Liquid Density g/cm ³
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀		
1	Before filling	6	0	85.615 0.011	11.723 0.011	1.387 0.011	0.543 0.005	0.733 0.009	1151.0 0.06 (42.876) (0.002)	0.4692 (113 K) 0.00002 0.4593 (120 K) 0.00002
	After filling	15	0	85.631 0.045	11.713 0.022	1.390 0.010	0.541 0.008	0.726 0.012	1150.77 0.68 (42.867) (0.024)	0.4691 (113 K) 0.0002 0.4592 (120 K) 0.0002
5	Before filling	6	1.240 0.0007	84.563 0.061	9.509 0.020	3.501 0.017	0.478 0.005	0.709 0.028	1151.6 1.1 (42.897) (0.040)	0.4788 (111 K) 0.0003 0.4662 (120 K) 0.0003
	After filling	25	1.242 0.006	84.556 0.023	9.527 0.019	3.498 0.007	0.480 0.002	0.697 0.003	1151.4 0.3 (42.891) (0.011)	0.4788 (111 K) 0.0001 0.4682 (120 K) 0.0001
6	Before filling	9	0.484 0.006	87.848 0.050	6.652 0.027	4.011 0.020	0.429 0.003	0.580 0.005	1141.2 0.6 (42.510) (0.022)	0.4537 (123 K) 0.0002
	After filling	39	0.478 0.003	87.823 0.027	6.658 0.016	4.025 0.014	0.431 0.002	0.584 0.006	1141.6 0.4 (42.525) (0.015)	0.4538 (123 K) 0.0001

*The day-to-day scatter due to calibration error has been subtracted from the estimated standard deviations presented in this and all of the following tables.

left open. Next, the analysis procedure was followed. The sample flow in the bypass line was uninterrupted during the purging procedure.

Initially, at least two analyses were made on each sample. Since each analysis took 14.5 minutes, this limited us to analyzing a maximum of two samples per hour. Once the integrator was repaired, only one analysis was made on each sample unless the results were more than three estimated standard deviations, for gas analysis alone, from the expected values. In this case, another analysis was made; in all cases, duplicate analyses were within one estimated standard deviation of the first analysis for all components. The prepared mixture from the cylinder was analyzed several times a day. Most often these were made during a temperature change and/or a LN₂ vessel filling -- each of which took about 30 minutes. The prepared mixture also was analyzed at the end of the day after shutting down the apparatus.

Sample flow to the Cook sampler was obtained by closing valves T6, T11, and T12 and by opening valves T7 and T8 and the valves on the Cook sampler (see figure 5.1). Once the sampler contained only liquid the valves on the sampler were closed and it was allowed to warm up. Next the accumulator was evacuated by closing valve T8 and opening valve T13 for several minutes. After closing valve T13, the downstream valve on the Cook sampler was opened. Then the accumulator was isolated by closing valve T7 and the mixture analyzed following the procedures described above.

5.1.2.4. Control of operating variables

The operating variables initially considered in this study were temperature, Reynold's number in the test section (Re), difference between the pressure at the sampling point and saturation pressure (Δp) and sampling rate (Q). The temperature was maintained by balancing the refrigerant flow against the heat input. Usually temperature control was held by having only valve N1 open (see figure 5.1); however, when operating at high sample flow rates, bypass valve N2 had to be opened as much as 1/4 turn. Both Re , i.e., flow rate in the test section, and Δp were set by adjusting pump speed and valve T2. (In all tests reported here valve T1 remained wide open.) Valve T15 controlled the sampling rate.

5.1.3. Experimental Results

The laboratory tests were divided into three phases:

- a) Probe Evaluation
- b) Vaporizer Evaluation
- c) General Tests.

The first two phases were conducted prior to the first flow facility tests. The last series of tests were made to explain the unexpectedly poor results obtained at the flow facility. The second flow facility tests and the shipboard tests followed the general laboratory tests.

5.1.3.1. Probe evaluation

The purpose of this phase of work was to:

- a) determine which, if any, operating variables affected the reliability of the three probes,
- b) determine which of the test probes was the most reliable,
- c) show that the reference probe produced samples of the same composition as the test probes, and
- d) establish the precision and accuracy of the reference probe.

The reason for using a reference probe was to determine the mixture composition. This probe design is unfeasible for most industrial applications. However, to prove the probe's reliability required tests using mixtures of known composition. In the probe evaluation three different compositions were used and two of these were mixtures of known composition.

In addition to the composition, the following operating variables were considered:

- a) three sample flow rates ($\dot{Q}$),
- b) four Reynold's numbers (Re) in the test section,
- c) two operating pressures, i.e., temperatures, and
- d) five differential pressures (Δp) i.e., the difference between the pressure at the sampling point and the saturation pressure of the mixture.

Table 5.2 lists the values of the variables according to run number and table 5.3 gives the estimated standard deviations in composition, ideal gas heating value and liquid density for each probe in each test. Except where noted in the text, average values of composition, ideal gas heating value, and liquid density for these tests are indistinguishable statistically from the corresponding values given in table 5.1. (Appendix 7.4 lists the raw data for all sampling tests.) Except for Test 2, the estimated standard deviation in heating value and density for the sampling data is presented as a percentage of the average values obtained from analyses of the prepared mixture in the gas cylinder. In Test 2 the percentage is based on the average of all of the probe analyses.

Test 1

The purpose of this test was to establish the feasible operating range of the variables and to identify operating variables which affect the reliability of the probes. Prepared mixture 1 was used in this test. Figure 5.2 shows the deviation in the computed ideal gas heating values as a function of probe and run number. The deviation is the difference between the heating value obtained via the probe sample and the heating value given in table 5.1 for mixture 1, all divided by the tabular value. Replicate points for a given run number represent the results of replicate gas analyses. We found that the occasional large scatter between replicate analyses was

Table 5.2. Date, run number and operating conditions used to evaluate the reference (R), side tap (S) and pitot tube (P) probes in conjunction with the reference vaporizer.

Date	Run Number	Temp.	Pressure, psia	Pressure, (kPa)	Probe	Δp , psi (kPa)	Sampling Rate slpm*	Reynolds Number
TEST 1.								
12/22	1	113.0	14.644	(100.97)	S	3.7 (25)	2.0	15800
"	2	"	"	"	R	"	"	"
"	3	"	"	"	P	"	"	"
"	4	"	"	"	P	"	6.0	"
12/23	5	"	"	"	S	"	"	"
"	6	"	"	"	R	"	"	"
"	7	"	"	"	P	"	2.0	"
"	8	"	"	"	P	2.7 (19)	"	"
"	9	"	"	"	P	1.8 (13)	"	"
"	10	"	"	"	P	"	"	26900
"	11	"	"	"	R	"	"	"
12/27	12	"	"	"	R	3.6 (25)	"	8700
"	13	"	"	"	S	"	"	"
"	14	"	"	"	P	"	"	"
"	15	"	"	"	P	"	6.0	"
"	16	"	"	"	S	"	"	"
"	17	"	"	"	R	"	"	"
12/28	18	120.0	25.072	(172.87)	R	"	4.6	20300
"	19	"	"	"	P	"	"	"
"	20	"	"	"	S	"	"	"
TEST 2.								
01/03	33	113.0	21.54	(148.5)	R	3.6 (25)	2.4	17500
"	34	"	"	"	P	"	"	"
"	35	"	"	"	S	"	"	"
"	36	"	"	"	R	"	"	"
"	37	"	"	"	S	"	"	"

* Slpm denotes standard liters per minute.

Table 5.2. (continued)

Date	Run Number	Temp.	Pressure, psia	Probe	Δp , psi (kPa)	Sampling Rate slpm	Reynolds Number
TEST 2. (continued)							
01/03	38	113.0	21.54	P	3.6 (25)	2.4	17500
01/04	39	"	" (148.5)	S	"	7.1	"
"	40	"	"	R	"	"	"
"	41	"	"	P	"	"	"
"	42	"	"	R	"	"	"
"	43	"	"	S	"	"	"
"	44	"	"	P	"	"	"
"	45	"	"	P	"	2.4	"
"	46	"	"	S	"	"	"
"	47	"	"	R	"	"	"
"	48	"	"	R	"	"	"
01/05	49	"	"	R	"	"	"
"	50	"	"	S	"	"	"
"	51	"	"	P	"	"	"
"	52	"	"	S	"	7.1	"
"	53	"	"	P	"	"	"
"	54	"	"	R	"	"	"
"	55	"	"	S	"	5.7	"
"	56	"	"	P	"	"	"
"	57	"	"	R	"	"	"
TEST 3.							
01/31	58	111.0	18.416	S	3.6 (25)	5.6	31500
"	59	"	" (126.97)	R	"	"	"
"	60	"	"	P	"	"	"
"	61	"	"	R	10.1 (70)	"	16600
"	62	"	"	S	"	"	"

Table 5.2. (continued)

Date	Run Number	Temp.	Pressure, psia	Probe	Δp , psi (kPa)	Sampling Rate slpm	Reynolds Number
TEST 3. (continued)							
01/31	63	111.0	18.416	P	10.1 (70)	5.6	16600
"	64	"	"	P	3.6 (25)	"	"
"	65	"	"	S	"	"	"
"	66	"	"	R	"	"	"
"	67	120.0	33.040	R	"	4.2	19900
"	68	"	"	P	"	"	"
"	69	"	"	S	"	"	"
02/01	70	"	"	S	"	"	37800
"	71	"	"	R	"	"	"
"	72	"	"	P	"	"	"
"	73	"	"	R	"	"	19900
"	74	"	"	S	"	"	"
"	75	"	"	P	"	"	"
"	76	"	"	R	10.1 (69)	"	"
"	77	"	"	S	"	"	"
"	78	"	"	P	"	"	"
02/02	79	111.0	18.344	S	3.6 (25)	5.6	16600
"	80	"	"	P	"	"	"
"	81	"	"	R	"	"	"
"	82	"	"	P	"	"	31500
"	83	"	"	S	"	"	"
"	84	"	"	R	"	"	"
"	85	"	"	P	10.1 (70)	4.2	16600
"	86	120.0	33.079	S	"	"	19900
"	87	"	"	R	"	"	"
02/03	88	"	"	S	"	"	"
"	89	"	"	R	"	"	"
"	90	"	"	P	"	"	"
"	91	"	"	S	3.6 (25)	"	37800

Table 5.2. (continued)

Date	Run Number	Temp.	Pressure, psia	Probe	ΔP , psi (kPa)	Sampling Rate slpm	Reynolds Number
TEST 3. (continued)							
02/03	92	120.0	33.079	P	3.6 (25)	4.2	37800
"	93	"	"	R	"	"	"
"	94	111.0	18.409	R	10.1 (70)	5.6	16600
"	95	"	"	S	"	"	"
"	96	"	"	P	"	"	"
TEST 4.							
02/25	146	111.0	18.310	R	4.0 (28)	5.6	1000
"	147	"	"	S	"	"	800
"	148	"	"	P	"	"	"
"	149	"	"	S	10.2 (70)	"	1300
"	150	"	"	R	"	"	1200
"	151	"	"	P	"	"	"
"	152	120.0	33.384	P	"	3.7	1200
"	153	"	"	S	"	"	1400
"	154	"	"	R	"	"	"
"	155	"	"	R	4.0 (27)	"	2000
"	156	"	"	P	"	"	1600
"	157	"	"	S	"	"	"
"	158	"	"	P	10.1 (70)	"	"
"	159	"	"	R	"	"	"
"	160	"	"	S	"	"	"
"	161	"	"	S	4.0 (28)	"	1800
"	162	"	"	P	"	"	"
"	163	"	"	R	"	"	"
"	164	111.0	18.317	R	10.1 (70)	5.6	1300
"	165	"	"	S	"	"	"

Table 5.2. (continued)

Date	Run Number	Temp.	Pressure, psia	Pressure, (kPa)	Probe	Δp , psi (kPa)	Sampling Rate slpm	Reynolds Number
TEST 4. (continued)								
02/25	166	111.0	18.317	(126.29)	P	10.1 (70)	5.6	1500
"	167	"	"	"	S	4.1 (28)	"	1300
"	168	"	"	"	R	"	"	"
"	169	"	"	"	P	"	"	"
02/28	170	"	"	"	P	"	"	1500
"	171	"	"	"	S	"	"	"
"	172	"	"	"	R	"	"	"
"	173	"	"	"	S	"	"	16500
"	174	"	"	"	R	"	"	"
"	175	"	"	"	P	"	"	"
"	176	"	"	"	R	"	"	1500
"	177	"	"	"	P	"	"	"
"	178	"	"	"	S	"	"	1700
"	179	"	"	"	S	"	"	16600
"	180	"	"	"	P	"	"	"
"	181	"	"	"	R	"	"	"

Table 5.3. Estimated standard deviations in composition, heating value and liquid density for probe tests 1 through 4.

Test Number	Variables	Probe ¹	Number of Points	Estimated Standard Deviation in							Heating ² Value, %	Liquid ² Density, %
				Composition, mol %								
				N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀			
1	Re, $\dot{Q}$, Δp , T	R	6	---	0.020	0.018	0.007	0.010	0.011	0.04	0.02	
		S	5	---	0.049	0.027	0.011	0.007	0.010	0.06	0.03	
		P	14	---	0.085	0.060	0.016	0.008	0.013	0.09	0.06	
2	$\dot{q}$	R	9	0.004	0.023	0.012	0.012	0.008	0.009	0.04	0.02	
		S	8	0.005	0.077	0.040	0.021	0.011	0.013	0.09	0.06	
		P	8	0.005	0.093	0.042	0.026	0.014	0.019	0.11	0.08	
3	Re, Δp , T	R	13	0.003	0.026	0.014	0.012	0.005	0.007	0.04	0.03	
		S	13	0.002	0.038	0.032	0.011	0.004	0.008	0.03	0.03	
		P	13	0.002	0.060	0.021	0.024	0.007	0.014	0.08	0.07	
4	Re, Δp , T	R	12	0.003	0.027	0.019	0.009	0.002	0.004	0.03	0.01	
		S	12	0.003	0.020	0.010	0.008	0.002	0.004	0.02	0.02	
		P	12	0.003	0.045	0.015	0.023	0.007	0.013	0.07	0.05	

¹ R denotes Reference, S denotes Side Tap, and P denotes Pitot Tube.

² These standard deviations are given as a percentage of the mean value as determined by analyses of the prepared mixture in the gas cylinder. However, the values for Run 2 are a percentage of the mean value of all probe analyses in the test.

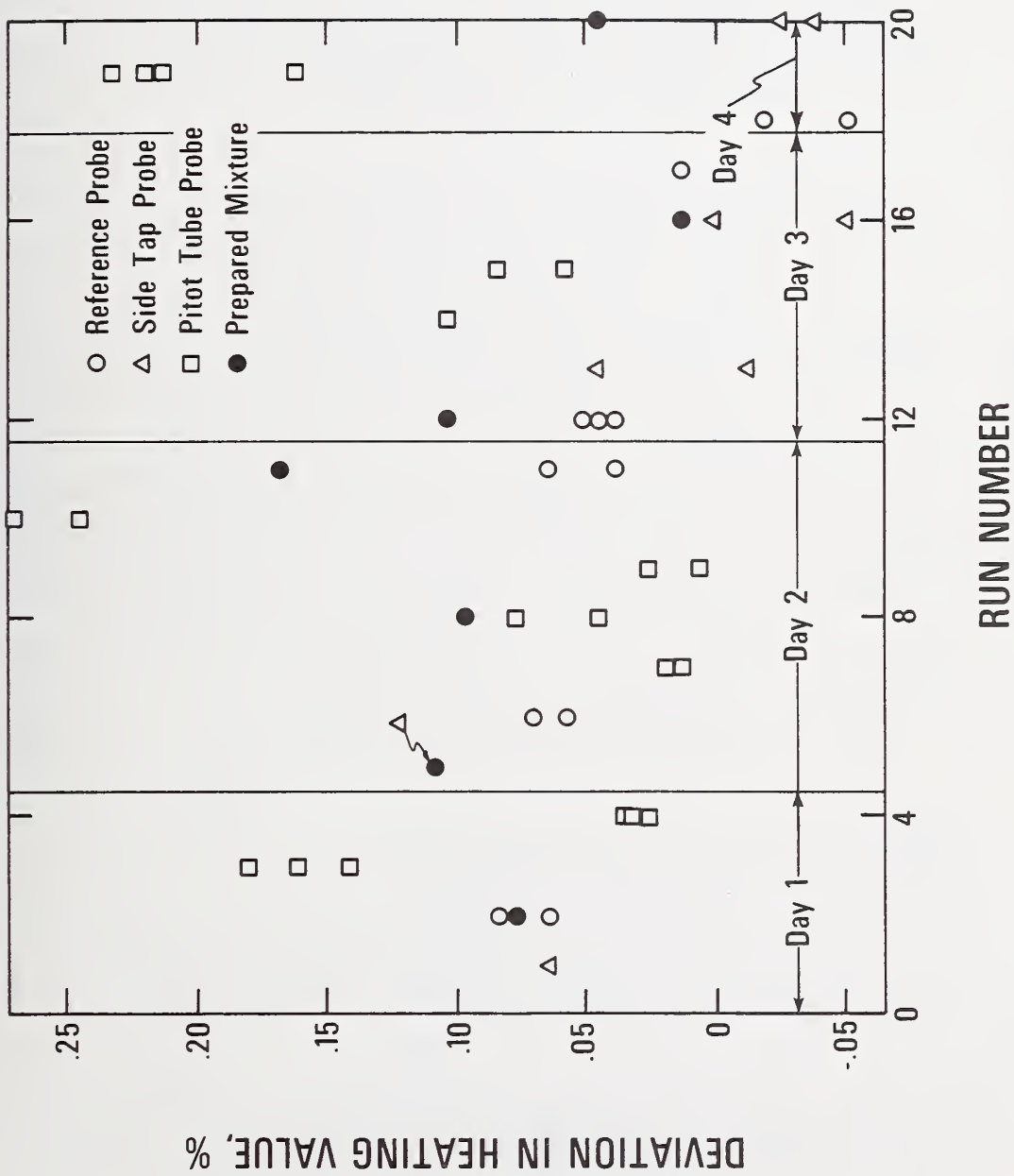


Figure 5.2. Deviations in the computed ideal gas heating values for probe test 1.

caused by a GC integrator malfunction (see section 4.2). Although this scatter confuses the results somewhat, figure 5.2 (and table 5.2) shows that the pitot tube tends to be more erratic. The results showed that it is possible to obtain representative samples at a Δp as low as 1.8 psi (0.012 MPa); the pressure drop in the sampling system prevented us from trying a lower Δp .

A statistical analysis of the results from the reference probe and the prepared mixture indicate that the reference probe collected a representative sample. The figure indicates that most of the computed heating values were slightly higher than the prepared mixture; this bias apparently was caused by not making three independent GC calibrations each day. Figure 5.2 shows that all three probes sampled the same liquid.

Test 2

The first test was not a statistically designed experiment; therefore, the effect of each operating variable could not be quantitatively determined. Nitrogen was added to the mixture to bring the nitrogen content to roughly 1.34 mol % for Test 2. (Two intermediate concentrations of 0.55 and 4.34 mol % nitrogen content were made before reaching the 1.34 mol % level; the data taken with these mixtures are given in Appendix 7.4 but not discussed here.) In this test the sampling rate was varied between 2.4 and 7.2 slpm (standard liters per minute, at 70° F (21° C) and 14.7 psia (0.101 MPa)). Figures 5.3 and 5.4 show the scatter, on a percentage basis about the mean, in the computed heating value and density as a function of run number and probe. There seems to be a day-to-day effect in the scatter with the first and last days being the worst and best, respectively. The only plausible explanation for this behavior is that the contents may not have been well mixed on the first day (this same trend seems apparent in figure 5.2).

In these tests the prepared mixture was condensed into the LNG vessel from the top. This unfortunately minimized the mixing during the filling process. The same procedure was followed when adding nitrogen. In each case the sampling tests began immediately after filling and the only mixing occurred during the recirculation of the LNG.

Table 5.4 lists the estimated standard deviations of the composition, heating value and liquid density as a function of probe and sampling rate. The test results show that the sampling rate has a strong effect on sampling precision.

Test 3

In this test we varied the temperature, flow rate and pressure difference but kept the sampling rate constant at 4.2 or 5.6 slpm. Prepared

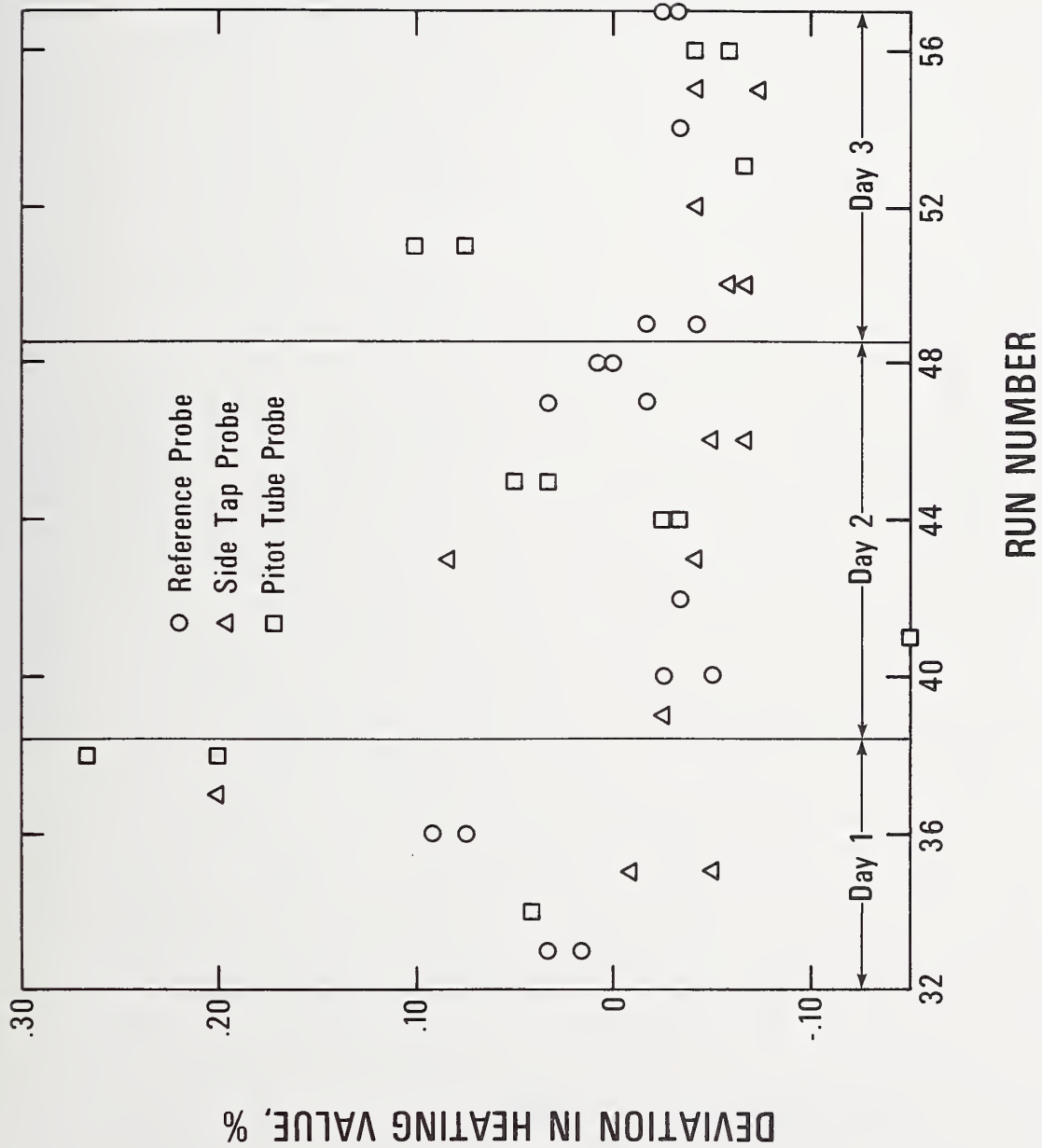
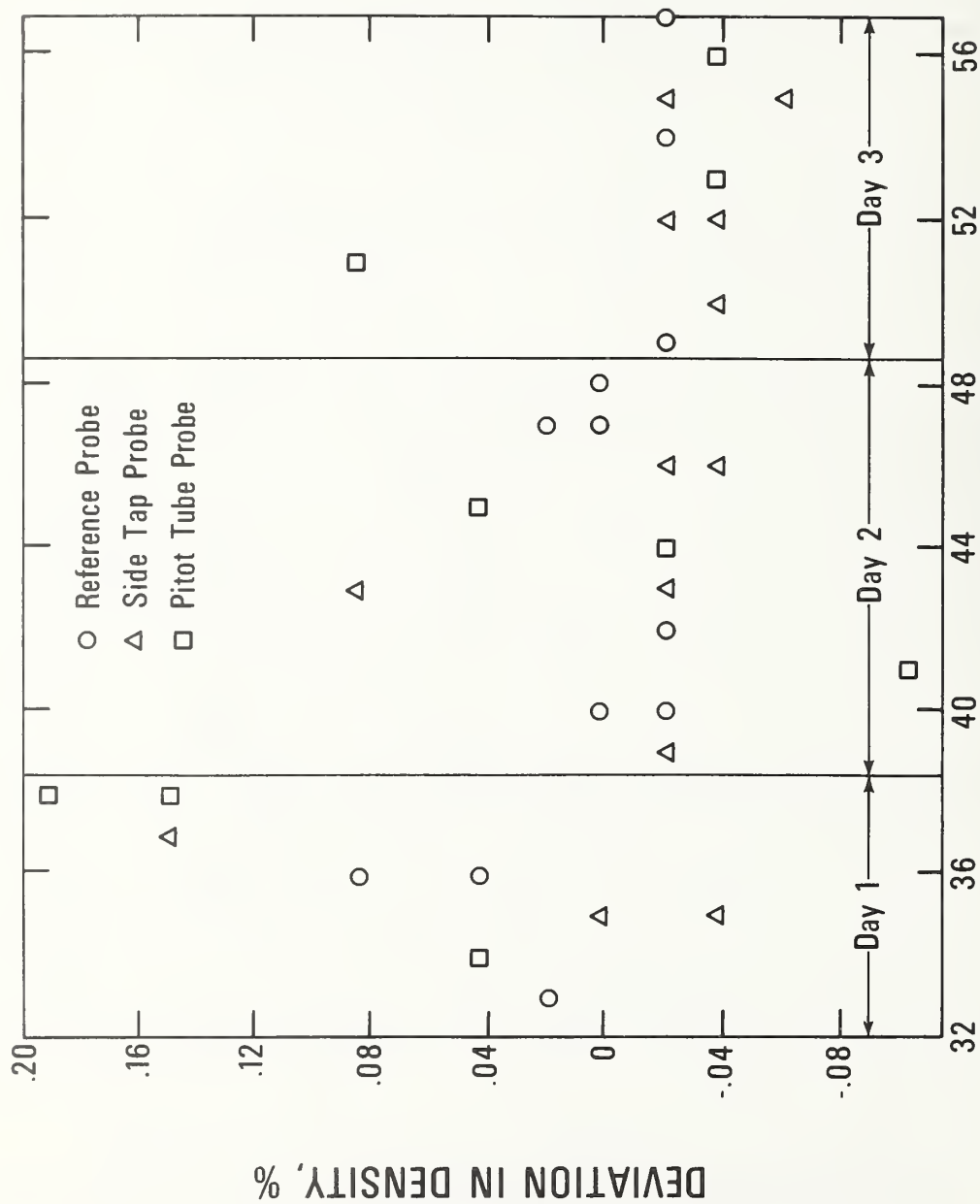


Figure 5.3. Deviations in the computed ideal gas heating values for probe test 2.



RUN NUMBER

Figure 5.4. Deviations in the computed liquid phase densities for probe test 2.

Table 5.4. Estimated standard deviations in composition, computed heating value and liquid density as a function of probe and sampling rate.

Sampling Rate slpm	Probe	Number of Points	Estimated Standard Deviation in								Heating Value %	Liquid Density %
			Composition, mol %					nC ₄ H ₁₀				
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀					
2.4	R	5	0.003	0.026	0.014	0.014	0.008	0.010	0.04	0.03		
	S	4	0.001	0.094	0.042	0.028	0.017	0.015	0.12	0.08		
	P	4	0.002	0.13	0.061	0.035	0.015	0.021	0.16	0.11		
7.2	R	4	0.002	0.006	0.011	0.007	0.004	0.003	0.007	0.00		
	S	4	0.004	0.016	0.011	0.006	0.003	0.007	0.016	0.02		
	P	4	0.003	0.013	0.006	0.006	0.002	0.007	0.015	0.01		

mixture 4 was used in this test. Recirculation pump characteristics permitted operation at both low and high flow rates (Reynolds numbers of 17,000 to 31,500) at a low Δp (3.6 psi (25 kPa)), but only the low flow rate at the high Δp (10.1 psi (70 kPa)). Shortly after starting this test the turbine meter stopped working. However, we had already noted the pump speed required for the desired flow rates at each Δp when operating at 111 K and used these to set the flow rate. We assumed that the same Δp and pump speed were required to obtain the desired flow rates at 120 K. This was verified later when the turbine meter began working again.

Figure 5.5 shows the deviation in the ideal gas heating value for this test. Neither the figure nor a statistical analysis of the data (see table 5.3) show an effect due to Re, Δp or T. Again, the pitot tube had the most scatter. The nitrogen content in the liquid was 0.024 mol% lower than in the prepared mixture 2; calculation showed that this could have been caused by nitrogen going into the vapor phase during condensation into the apparatus.

Test 4

The purpose of this short test was to ascertain the effect of laminar flow on sampling precision. The difference in operating variables between this test and test 3 was that the Reynold's number was 1500 or 17,000 instead of 17,000 or 31,500. In this test the low flow rate was maintained the first day while Δp and temperature were varied; the second day the flow rate and Δp were varied while the temperature was held constant at 111 K. Figure 5.6 shows the deviation in ideal gas heating value. During the first part of the second day the power supply for the pump frequently tripped the circuit breaker. Each shutdown caused the vaporizer to warm up. We believe that the increased scatter during the first part of day 2 was caused by the reference vaporizer not being at steady state conditions when the gas sample was trapped in the accumulator. Once the power supply problem was fixed (prior to run number 177) the scatter became comparable to that of the previous day.

The results indicate that the probes work equally well in laminar and turbulent flow. The statistical analysis shows that the nitrogen content decreased by 0.006 mol% when going from 111 to 120 K. This effect had not been detected in any other test, nor for any other component. However, by the time of this test roughly 100 samples had been withdrawn from the apparatus, thus increasing the vapor space in the LNG vessel. Calculations showed that this apparent temperature effect could have been caused by the increased amount of nitrogen in the vapor phase at 120 K.

5.1.3.2. Vaporizer evaluation

The probe tests showed that the reference probe to be the most reliable of the three probes tested. Therefore, the reference probe was used in the evaluation of vaporizer A and the Cook sampler. Since the Cook sampler is

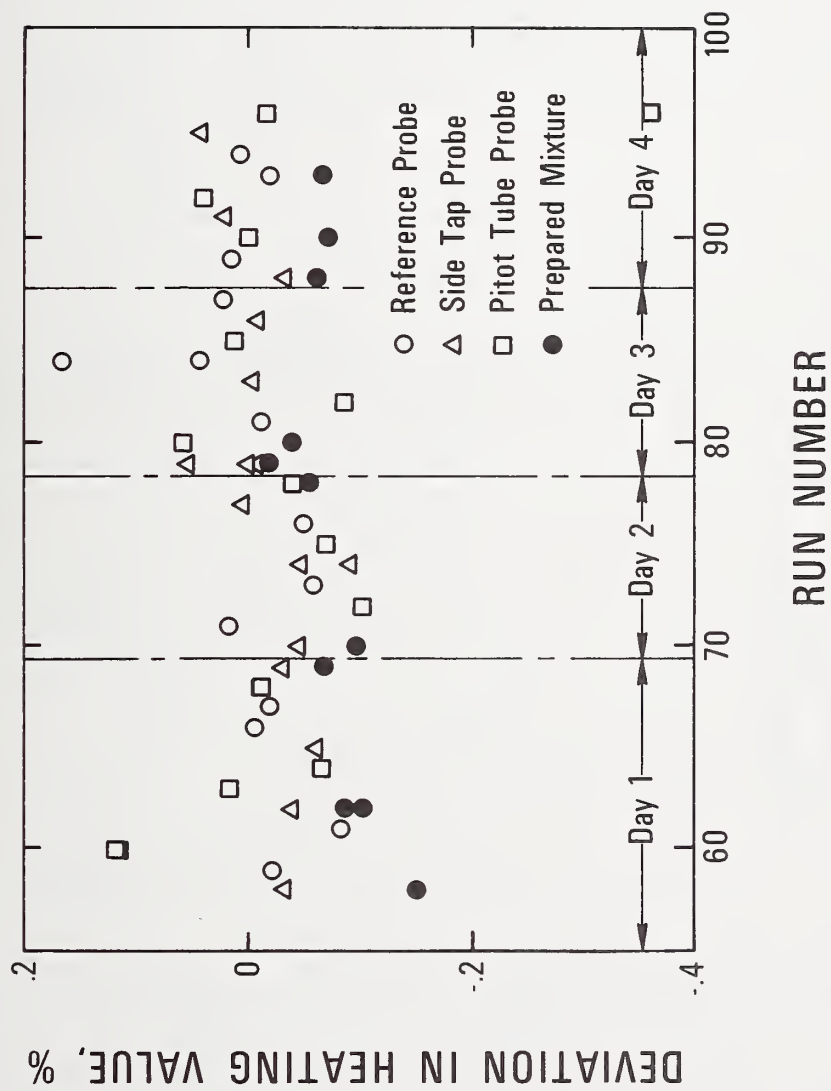
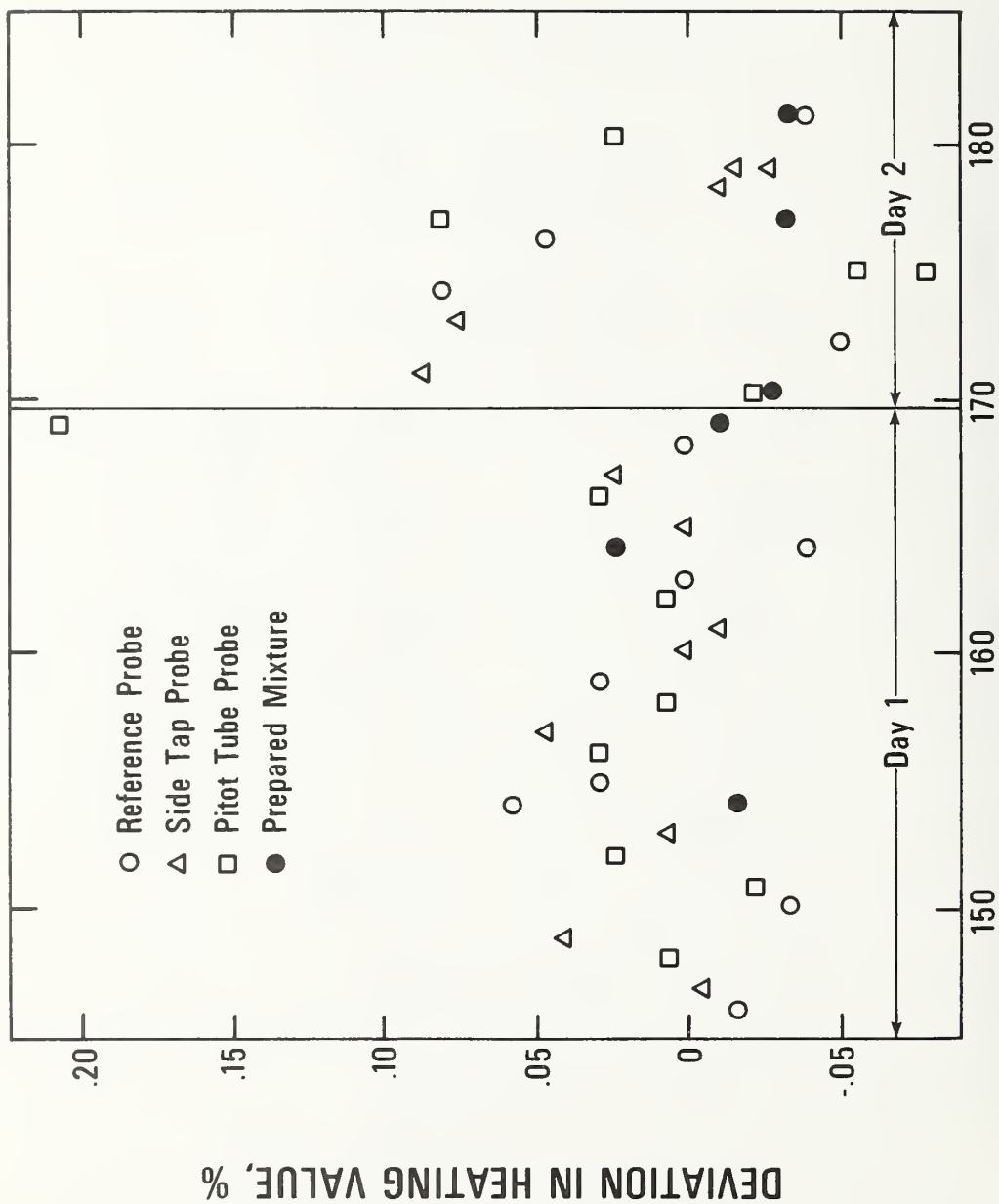


Figure 5.5. Deviations in the computed ideal gas heating values for probe test 3.



RUN NUMBER

Figure 5.6. Deviations in the computed ideal gas heating values for probe test 4.

is designed for batch sampling, it is not a continuous vaporizer like the reference vaporizer and vaporizer A. Preliminary testing of the Cook sampler showed that it would not work well on the laboratory apparatus. Cook [22] indicated that it is designed for use with high sampling rates so that the total sampling time would be less than 30 seconds. In our tests the sampling rate was so low that it took at least 30 minutes to cool the sampler down. By this time the sampler valves were very cold and leaked when closed. Because of this problem and because the primary interest was in continuous sampling systems the Cook sampler was not tested further on the laboratory apparatus. However, vaporizer A was found to work at low sampling rates; it was evaluated in the same manner as the probes. Prepared mixture 4 was used in both tests. Table 5.5 lists the values of the operating variable and 5.6 lists the estimated standard deviation in composition, ideal gas heating value and liquid density.

Test 1

The purpose of this test was to evaluate vaporizer A at two sampling rates (2.0 and 5.6 slpm), temperatures (111 and 120 K), pressure differences (3.6 to 10.1 psi (25 to 70 kPa)) and flow rates (Re of 18,000 to 35,000); these conditions are comparable to those used for the probe evaluations. Figure 5.7 shows the deviation in heating values for this test. The estimated standard deviation in the heating value for this vaporizer was about 20 times greater than that observed for the reference vaporizer during the probe tests. However, the precision of the reference system was poorer in this test than in any other test. This increased scatter was due to either incomplete accumulator purging and/or to taking a sample too quickly after starting the reference vaporizer. Three points on the third day showed that the reference system precision was a factor of three worse than usual. During these runs the accumulator residence time was 40 to 120 seconds; the samples were analyzed after 10 minutes of purging the accumulator. The fourth reference sample was taken after the accumulator was purged for 20 minutes this sample was representative of the prepared mixture.

Once it was obvious that vaporizer A was not performing well, we made several minor changes to try to reduce the scatter. Originally the sampling rate was set by using valve T15 (see figure 5.1); starting with run 137, the sample rate was set by adjusting the pressure regulator on the vaporizer. This did not improve the results. During most of this test the throttling valve (valve T6, figure 5.1) for the reference vaporizer was roughly 1/4 open. We made two reference vaporizer runs (runs 108 and 109) with the valve wide open, thinking that the valve might be preventing backflashing (this was the purpose of the valve). With the valve wide open, the pressure drop in the lines between the probe and the two vaporizers was comparable.

Table 5.5. Date, run number and operating conditions used to evaluate the reference (R) vaporizer and vaporizer A (A); reference probe used throughout vaporizer tests.

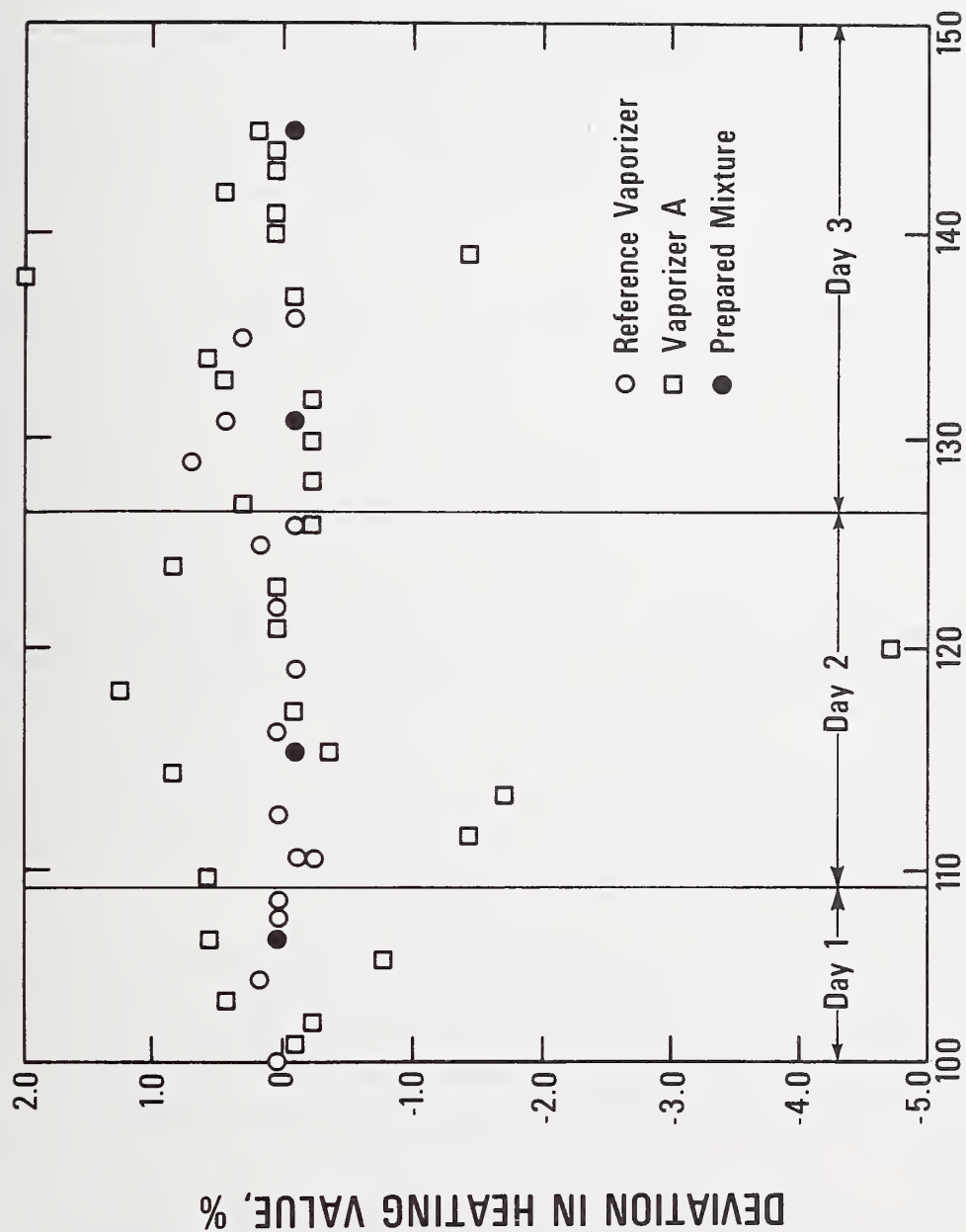
Date	Run Number	Temp. K	Pressure, psia	Pressure, (kPa)	Vaporizer	Δp , psi	Δp , (kPa)	Sampling Rate slpm	Reynolds Number
TEST 1.									
02/22	100	111.0	18.492	(127.50)	R	3.7	(25)	5.6	33100
"	101	"	"	"	A	"	"	"	"
"	102	"	"	"	A	10.1	(70)	"	8500
"	103	"	"	"	A	3.6	(25)	2.0	17000
"	104	"	"	"	R	"	"	5.6	"
"	105	"	"	"	A	"	"	"	"
"	106	"	"	"	A	"	"	2.0	33200
"	107	"	"	"	R	10.2	(70)	"	16400
"	108	"	"	"	R	"	"	"	"
02/23	109	120.0	33.264	(229.35)	A	3.6	(25)	4.2	20300
"	110	"	"	"	R	"	"	"	"
"	111	"	"	"	A	"	"	1.5	41000
"	112	"	"	"	R	10.1	(70)	"	20400
"	113	"	"	"	A	"	"	"	"
"	114	"	"	"	A	"	"	4.0	"
"	115	"	"	"	A	3.6	(25)	"	41100
"	116	"	"	"	R	"	"	"	"
"	117	"	"	"	A	"	"	1.5	20300
"	118	111.0	18.492	(127.50)	A	10.1	(70)	"	17700
"	119	"	"	"	R	"	"	"	"
"	120	"	"	"	A	3.6	(25)	"	34200
"	121	"	"	"	A	6.5	(45)	5.6	17500
"	122	"	"	"	R	3.6	(25)	"	"
"	123	"	"	"	A	"	"	"	"
"	124	"	"	"	A	"	"	2.0	"
"	125	"	"	"	R	"	"	5.5	33400
"	126	"	"	"	A	"	"	"	"
02/24	127	120.0	33.218	(229.03)	A	"	"	1.5	42200
"	128	"	"	"	A	10.1	(70)	4.2	20200
"	129	"	"	"	R	"	"	1.5	"

Table 5.5. (continued)

Date	Run Number	Temp. K	Pressure, psia	Pressure, (kPa)	Vaporizer	Δp , psi	Δp , (kPa)	Sampling Rate slpm	Reynolds Number
TEST 1. (continued)									
02/24	130	120.0	33.218	(229.03)	A	10.1	(70)	1.5	20200
"	131	"	"	"	R	"	"	"	"
"	132	"	"	"	A	3.6	(25)	4.1	41200
"	133	"	"	"	A	"	"	"	20200
"	134	"	"	"	A	"	"	1.4	"
"	135	120.0	33.218	(229.03)	R	3.6	(25)	4.1	20200
"	136	"	"	"	R	"	"	"	41400
"	137	"	"	"	A	10.1	(70)	10.7	20800
"	138	"	"	"	A	"	"	4.1	"
"	139	"	"	"	A	"	"	1.8	"
"	140	"	"	"	A	3.6	(25)	10.7	41200
"	141	"	"	"	A	"	"	"	20100
"	142	111.0	18.513	(127.64)	A	"	"	10.0	16800
"	143	"	"	"	A	"	"	"	33900
"	144	"	"	"	A	10.2	(70)	"	16900
"	145	"	"	"	A	3.6	(25)	"	"
TEST 2.									
03/01	182	111.0	18.26	(125.9)	A	4.8	(33)	10.1	16400
"	183	"	"	"	A	"	"	6.6	"
"	184	"	"	"	A	3.9	(27)	"	33000
"	185	"	"	"	A	"	"	9.9	"
"	186	"	"	"	A	"	"	6.6	16600
"	187	"	"	"	A	"	"	10.1	"
"	188	"	"	"	A	"	"	"	33200
"	189	"	"	"	A	"	"	6.6	"
"	190	"	"	"	R	"	"	"	"

Table 5.6. Estimated standard deviation in composition, computed ideal gas heating value and liquid density for vaporizer tests 1 and 2 using reference vaporizer (R) and vaporizer A (A).

Test	Vaporizer	Number of Points	Estimated Standard Deviation in								Heating Value %	Liquid Density %
			Composition, mol %									
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	cC ₄ H ₁₀	nC ₄ H ₁₀				
1	A	29	0.016	0.60	0.35	0.30	0.066	0.11	0.76	0.80		
	R	10	0.004	0.090	0.054	0.044	0.008	0.011			0.11	0.06
2	A	8	0.021	0.18	0.11	0.065	0.024	0.047	0.25	0.19		



RUN NUMBER

Figure 5.7. Deviations in the computed ideal gas heating values for vaporizer test 1.

Having the valve wide open did not diminish the performance of the reference probe. Near the end of the test the sampling rate was increased to 16 slpm (runs 137, 140 and 141) or 10 slpm (the last five runs). Since the higher sampling rate seemed to improve the vaporizer performance, another test was planned to determine the effect of high sampling rate.

Test 2

In this test we operated at 111 K and a Δp of roughly 3.8 psi (27 kPa) and varied the Reynold's number between 16,000 and 33,000 and sampling rate between 6.7 and 19 slpm. Figure 5.8 shows the deviation in the heating values for this test. The estimated standard deviation in heating value for vaporizer A in this test was 0.25 percent as compared to 0.76 percent for the first test but still high compared to the typical 0.05 percent obtained for the reference vaporizer. We do not know why runs 188 and 189 are so different from the rest of the data.

5.1.3.3. General tests

To explain the poor results obtained in the first flow facility test (see section 5.2.1.1) it was necessary to conduct additional laboratory measurements. These tests considered whether sampling precision could be affected by:

- a) eliminating the sample accumulator,
- b) increasing or decreasing the vaporizer outlet temperature,
- c) heat leak to the sample probe,
- d) minimizing the pressure drop upstream of the vaporizer,
- e) decreasing the sample residence time in the accumulator, and
- f) having relatively high concentrations of heavy hydrocarbons (C_5 's through C_8 's) in the LNG.

The following operating variables were held constant during each test:

- a) temperature and pressure,
- b) sampling rate, $\dot{Q}$ (except when varying accumulator residence time),
- c) Reynold's number (Re) in the test section, and
- d) differential pressure, Δp .

Table 5.7 gives the values of the operating variables for these tests. These variables values were in the range known to give good results in the previous laboratory tests. Prepared mixture 6 (see table 5.1) was used in tests 1 through 5; LNG, which was prepared by condensing natural gas containing C_6+ was used in the last test. The reference vaporizer was used in all of the tests.

Test 1

The purpose of this test was to determine the sampling precision when drawing samples from the accumulator bypass line while having the accumulator bypassed. In all previous laboratory tests (but not the first flow facility test) samples had been taken only from the accumulator. Table 5.8 and

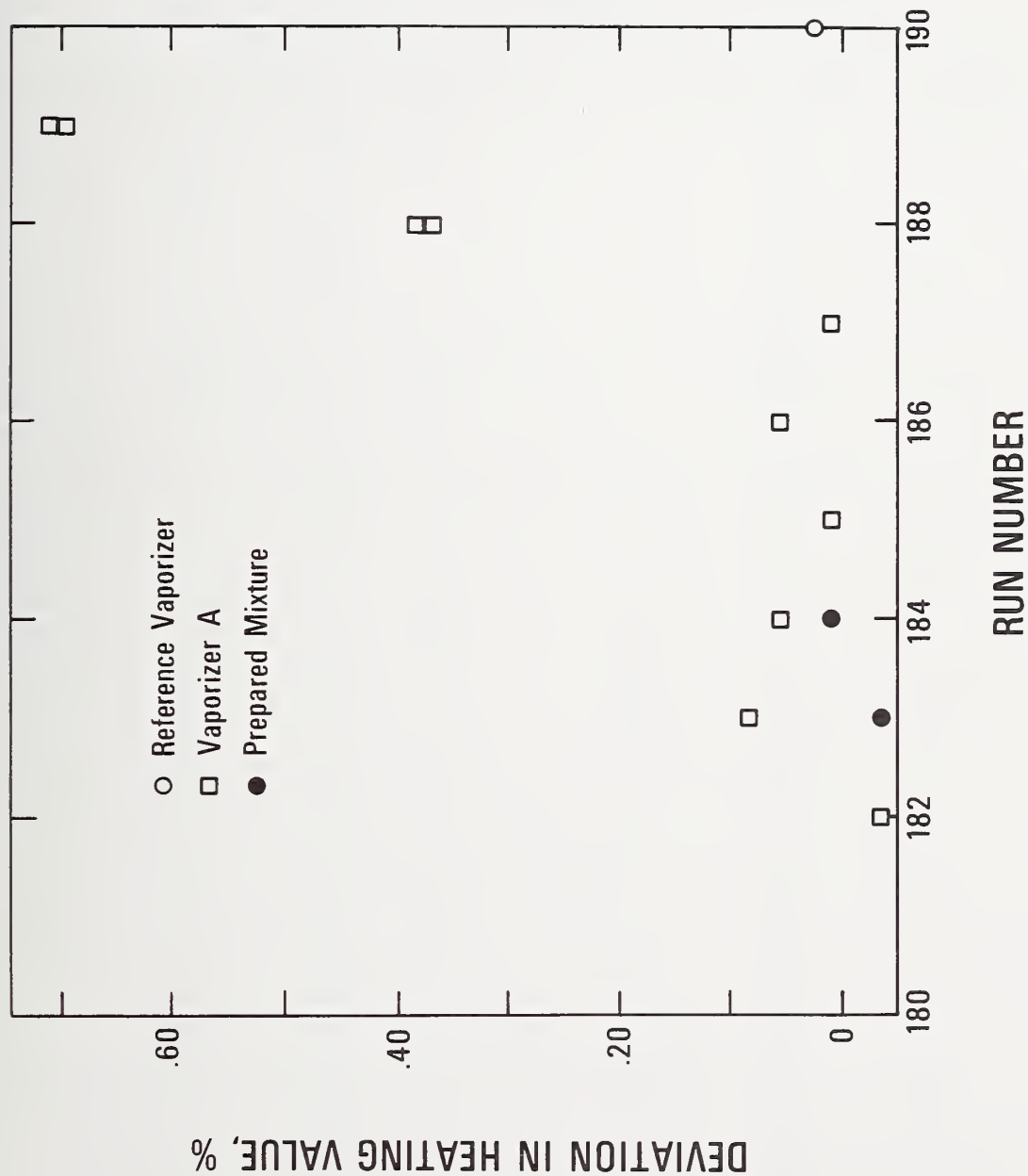


Figure 5.8. Deviation in the computed ideal gas heating values for vaporizer test 2.

Table 5.7. Date, run number and operating conditions for general tests; reference vaporizer used throughout these tests; R, S, and P refer to reference, side tap and pitot tube probes, respectively.

Test 1. Temperature = 123.0 K, Pressure = 36.0 psia (248 kPa), Sampling Rate = 5.0 slpm, Reynolds Number = 22,800, ΔP = 5.0 psi (35 kPa).

Date	Run Number	Probe	Accumulator Bypassed
4/14	191	R	No
"	192	"	Yes
"	193	"	Yes
"	194	"	No
"	195	"	Yes
"	196	"	Yes
"	197	"	No
"	198	P	No
"	199	"	Yes
"	200	"	Yes
4/15	201	"	No
"	202	R	No
"	203	"	Yes
"	204	S	No
"	205	"	Yes
"	206	P	No
"	207	"	Yes
"	208	S	No
4/18	209	R	Yes
"	210	S	Yes
"	211	P	Yes
"	212	"	No
4/19	213	R	No
"	214	"	Yes
"	215	S	Yes
"	216	"	No
"	217	P	Yes
"	218	"	No
"	219	S	No
"	220	"	Yes
"	221	P	Yes
"	222	"	No
"	223	R	Yes
"	224	"	No
"	225	P	No
"	226	"	Yes
4/20	227	R	Yes
"	228	"	No
"	229	S	Yes
"	230	"	No

Table 5.7. (Continued)

Test 1. (Continued)

Date	Run Number	Probe	Accumulator Bypassed
4/20	231	R	Yes
"	232	"	No
"	233	P	Yes
"	234	"	No
"	235	S	Yes
"	236	"	No
"	237	"	Yes
"	238	"	Yes

Test 2. Temperature = 123.0 K, Pressure = 36.0 psia (238 kPa), Sampling Rate = 5.0 slpm, Reynolds Number = 22,800, ΔP = 5.0 spi (36 kPa), Probe = side tap.

Date	Run Number	Vaporizer Power, Watts	Accumulator Bypassed
4/21	239	0.0	Yes
"	240	"	No
"	241	"	Yes
"	242	"	No
"	243	"	Yes
"	244	"	No
"	245	99.6	No
"	246	98.6	Yes
"	247	97.5	No
"	248	98.1	Yes
"	249	"	Yes
"	250	"	No
4/22	251	0.0	No
"	252	"	Yes
"	253	"	No
"	254	"	Yes
"	255	"	No
"	256	"	Yes
"	257	98.6	No
"	258	97.5	Yes
"	259	97.0	No
"	260	"	Yes
"	261	96.5	No
"	262	"	Yes
4/25	263	63.2	No
"	264	62.0	No

Table 5.7. (Continued)

Test 3. Temperature = 123.0 K, Pressure = 35.5 psia (245 kPa), Sampling Rate = 5.0 slpm, Reynolds Number = 22,900, Δp = 5.0 psi (35 kPa).

Date	Run Number	Probe	Probe Heater Power Watts
4/25	265	S	0.1
"	266	"	0.4
"	267	"	1.7
"	268	"	6.7
4/26	269	P	0.0
"	270	"	0.1
"	271	"	0.4
"	272	"	1.0
"	273	"	1.7
"	274	"	3.8
"	275	"	6.7
4/29	299	"	0.0
"	300	"	1.7
"	301	"	6.7
"	302	"	1.7
"	303	"	6.7
"	304	"	0.0
"	305	"	6.7
"	306	"	1.7
"	307	"	0.0
"	308	"	1.7
"	309	"	0.0
"	310	"	6.7

Test 4. Temperature = 123.0 K, Pressure = 35.3 psia (243 kPa), Sampling Rate = 5.0 slpm, Reynolds Number 23,500, Δp = 5.0 psi (35 kPa), Probe = side tap.

Date	Run Number	Needle Valve, Throttled
4/26	276	Yes
"	277	Yes
"	278	No
"	279	Yes
"	280	No
4/27	281	No
"	282	Yes
"	283	No
"	284	Yes
"	285	No
"	286	Yes
"	287	No
"	288	Yes
"	298	No
"	299	Yes

Table 5.7. (Continued)

Test 4. (Continued)

Date	Run Number	Needle Valve, Throttled
4/27	291	No
"	292	Yes
5/02	311	No
"	312	No
"	313	No
"	314	No
"	315	No
"	316	No
"	317	Yes
"	318	Yes
"	319	Yes
"	320	Yes
"	321	Yes

Test 5. Temperature = 112.0 K, Pressure = 16.8 psi (116 kPa), Δp = 8 psi, Reynolds Number = 9,100 (56 kPa), Probe = side tap.

Date	Run Number	Sampling Rate slpm	Accumulator Residence Time, s
4/28	293	12.2	10.5
"	294	8.6	15.0
"	295	5.9	21.8
"	296	12.2	10.5
"	297	8.6	15.0
"	298	12.2	10.5

Test 6. Sampling Rate = 5 slpm, Δp = 5.0 psi (35 kPa), Reynolds Number = 22,000.

Date	Run Number	Temperature K	Pressure psia kPa		Probe
5/11	322	123.5	32.2	222	S
"	323	"	"	"	"
"	324	"	"	"	"
"	325	"	"	"	"
"	326	"	"	"	"
"	327	"	"	"	"
5/12	328	121.5	32.1	221	"
"	329	"	"	"	"
"	330	"	"	"	"
"	331	"	"	"	"

Table 5.7. (Continued)

Test 6. (Continued)

Date	Run Number	Temperature K	Pressure		Probe
			psia	kPa	
5/12	332	121.5	32.1	221	S
"	333	"	"	"	"
"	334	"	"	"	"
5/16	335	120.0	40.1	276	"
"	336	"	"	"	"
"	337	"	"	"	P
"	338	"	"	"	"
"	339	"	"	"	"
"	340	"	"	"	"
"	341	"	"	"	"

Table 5.8. Estimated standard deviation in the composition, heating value and liquid density for the three probes when samples were drawn from the accumulator and from the bypass line.

Sampling Point	Probe	Number of Points	Estimated Standard Deviation in							Heating Value %	Liquid Density %
			Composition, mol %								
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀		iC ₄ H ₁₀		
Accumulator	R	8	0.0009	0.025	0.012	0.014	0.003	0.003	0.03	0.02	
	S	8	0.0004	0.021	0.029	0.001	0.002	0.004	0.02	0.009	
	P	6	0.0005	0.017	0.024	0.028	0.003	0.004	0.05	0.04	
Bypass	R	10	0.008	0.24	0.21	0.063	0.008	0.014	0.18	0.15	
	S	8	0.030	1.8	0.95	0.69	0.089	0.126	2.01	1.61	
	P	8	0.016	0.30	0.29	0.073	0.019	0.031	0.26	0.20	

figure 5.9 show that the scatter increased markedly when the accumulator was not used. This indicates that the vaporization process generates inhomogeneities in the sample which can be eliminated by time averaging the sample in an accumulator. The accumulator was used in all remaining tests.

In addition to taking discrete samples we continuously fed a small gas sample stream (~ 10 cc/min) to an ultrasonic detector. It was hoped that by continuously monitoring overall composition fluctuations (there was no easy way to selectively monitor one component) a periodic behavior could be found. Samples from the bypass line, accumulator, prepared mixture cylinder and from a pure methane cylinder were analyzed continuously. Only the pure methane sample showed no composition fluctuations. The other samples showed random fluctuations with the bypass sample having the largest scatter. The fluctuations in the prepared mixture and accumulator samples were comparable in magnitude. This indicates that even gas mixtures withdrawn from cylinders will contain some composition variations. Unfortunately, it was not possible to quantify the fluctuations in terms of mol percent.

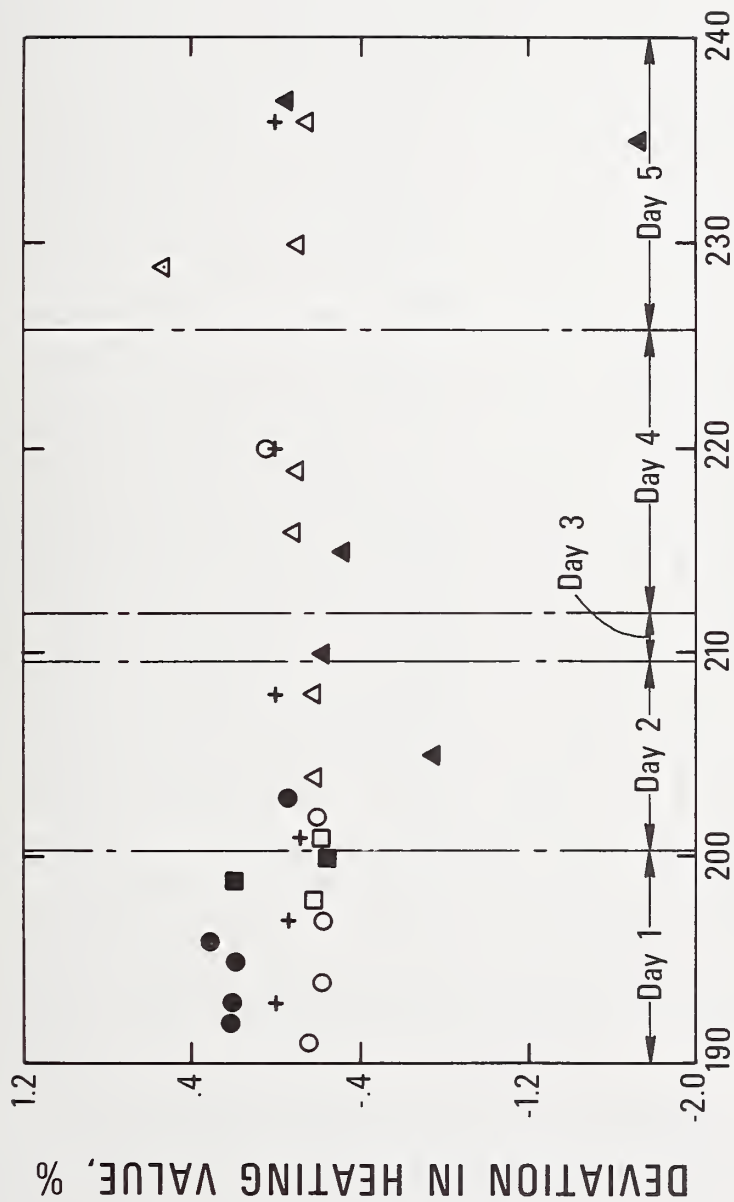
Test 2

The dew point temperature of the LNG mixtures in the first flow facility test was well below ambient temperature; however, it was thought that a possible problem could have been the use of a long vaporizer warmed only by cold water. To simulate this condition in the laboratory we varied the power to the vaporizer; this, in effect, changed the vaporizer length. Three different power levels, 0, 62 and 98 watts, were used. With no input power, the vaporizer plus approximately 5 feet (1.5 m) of the 1/4 inch O.D. (6.4 mm) copper tubing downstream of the vaporizer became coated with frost. At 62 watts the vaporizer outlet temperature was warm to the touch; this was the temperature that was used in all other laboratory tests. At 100 watts, the tubing just downstream of the vaporizer was too hot to touch for more than a second. At all three power levels the accumulator remained at ambient temperature. Only the side tap probe was used in this test and the values for the 62 watt case were taken from the previous test.

Table 5.9 shows that only the 0 power case affected the sampling precision. These results tend to indicate that it is better to vaporize the sample quickly. It was thought that the higher heat input might offset the effect of bypassing the accumulator. However, table 5.9 and figure 5.10 show that the increased heat input did not improve the sampling precision when the accumulator was bypassed.

Test 3

In the first flow facility test the lines from the probes to the vaporizers were uninsulated. Therefore, it was thought that heat leak could cause the observed sampling problems. To test the effect of heat leak we

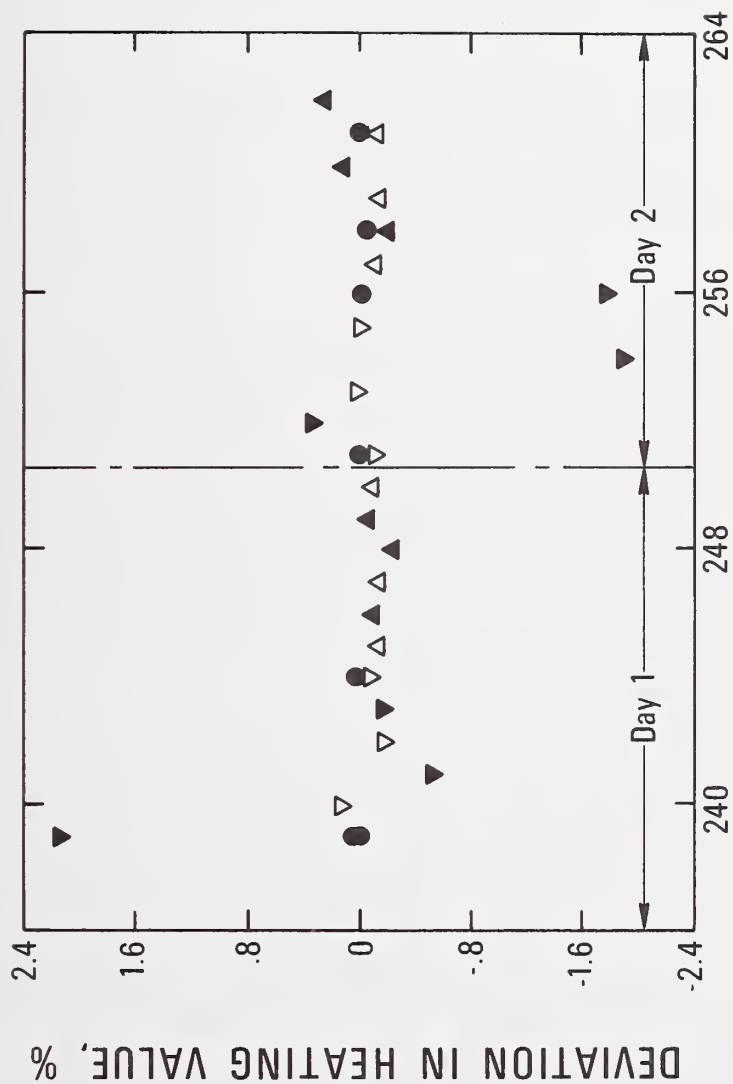


RUN NUMBER

Figure 5.9. Deviations in the computed ideal gas heating values for general test 1. The symbols ○, Δ, and □ denote samples taken using the reference, side tap and pitot tube probes, respectively, and the accumulator. The symbols ●, ▲, and ■ denote samples taken using the reference, side tap and pitot tube probes with the accumulator bypassed. The symbol + denotes analysis of the prepared mixture.

Table 5.9. Estimated standard deviation in the composition, heating value and liquid density for the side tap probe with different heat inputs to the reference vaporizer.

Heater Input Power, Watts	Number of Points	Estimated Standard Deviation in Composition, mol %							Heating Value %	Liquid Density %
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀			
0	6	0.018	0.095	0.034	0.054	0.010	0.016	0.12	0.10	
62	12	0.001	0.018	0.013	0.014	0.002	0.004	0.02	0.02	
98 (with accumulator)	6	0.002	0.020	0.015	0.008	0.003	0.003	0.02	0.02	
98 (accumulator bypassed)	5	0.006	0.18	0.12	0.068	0.006	0.008	0.18	0.14	



RUN NUMBER

Figure 5.10. Deviations in the computed ideal gas heating values for general test 2. The symbols Δ and $\blacktriangle$ denote 100 watt power to the vaporizer with and without using the accumulator, respectively. The symbols ∇ , $\blacktriangledown$ and $\bullet$ denote 0 power input, with and without the accumulator and prepared mixture analysis, respectively.

attached 42 ohm heaters to the pitot and side tap probe. Since the probe line was 1/16 inch (1.6 mm) O.D. tubing it was impractical to wrap heater wire directly on the tubing. The heater wire was wrapped on a copper cylinder which was soft soldered to the probe lines approximately 1.5 inches (38 mm) from where they entered the test section. The heaters covered approximately 0.5 inches (13 mm) of the 1/16 inch (1.6 mm) stainless steel tube giving a heat transfer area of 0.10 in^2 (63 mm^2).

In the first part of the test we heated the side tap and took samples at input powers of 0.10, 0.42, 0, 1.7 and 6.7 watts -- this corresponds to 0.2, 0.7, 0, 3 and 11 percent of the power (62 watts) to the vaporizer. As figure 5.11 shows there was no apparent effect on sampling. At 6.7 watts the heater shorted out. The test was repeated for the pitot tube. Although, statistically, there was no apparent change in the precision, increasing the heat seemed to decrease the methane and nitrogen content (runs 269 through 275 in figure 5.12). Therefore, it was decided to repeat the test with the heat input randomized instead of systematically increasing the input power. Table 5.10 lists the estimated standard deviations. This test showed that the apparent trend noted before for nitrogen was coincidence and that there was no detectable effect on the sampling precision.

During the last run of this test, this heater shorted out too. When the apparatus was disassembled we found that both heaters were unsoldered from the tubing. Soft solder (50% Sn, 50% Pb) melts at 490 K [23] and the thermal conductivity for 304 stainless steel is roughly 0.12 watts/cm-K [24]. Using these values, the heater length and tube dimension, and 6.7 watts input power, thermal conductivity calculations show that the temperature of the inside tube wall could have been as high as 480 K. Therefore, there had to be film boiling inside the probe line. The effect of heat leak was studied in the second flow facility test.

Test 4

During previous tests the needle valve upstream of the reference vaporizer was used to provide some pressure drop in the liquid phase which would reduce the possibility of backflashing. In the vaporizer evaluation (see section 5.1.3.2) we opened the needle valve and found no apparent change in the sampling precision. If the needle valve could be omitted it would eliminate an operating variable in the LNG sampling system. Therefore, we wanted to confirm the previous results in this test using the side tap instead of the reference probe.

When wide open there was an estimated 0.03 psi (0.2 kPa) across the needle valve (valve T6, figure 5.1); in this case regulating valve T15 was used to control the sample flow rate. The maximum pressure drop (~ 1.5 psi (10.3 kPa)) across the needle valve was obtained by using it to control the sampling rate; in this case all valves downstream of the needle valve were wide open. (We estimated the pressure drop in the vaporizer and the line to

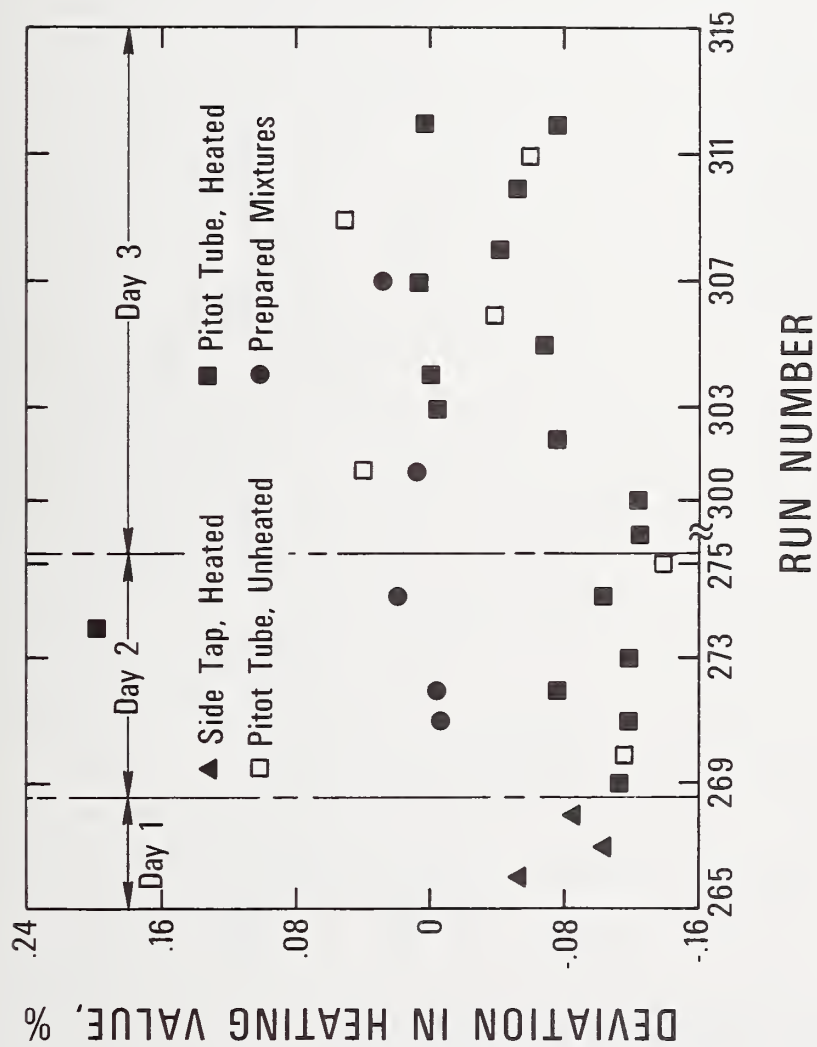


Figure 5.11. Deviations in the computed ideal gas heating values for general test 3.

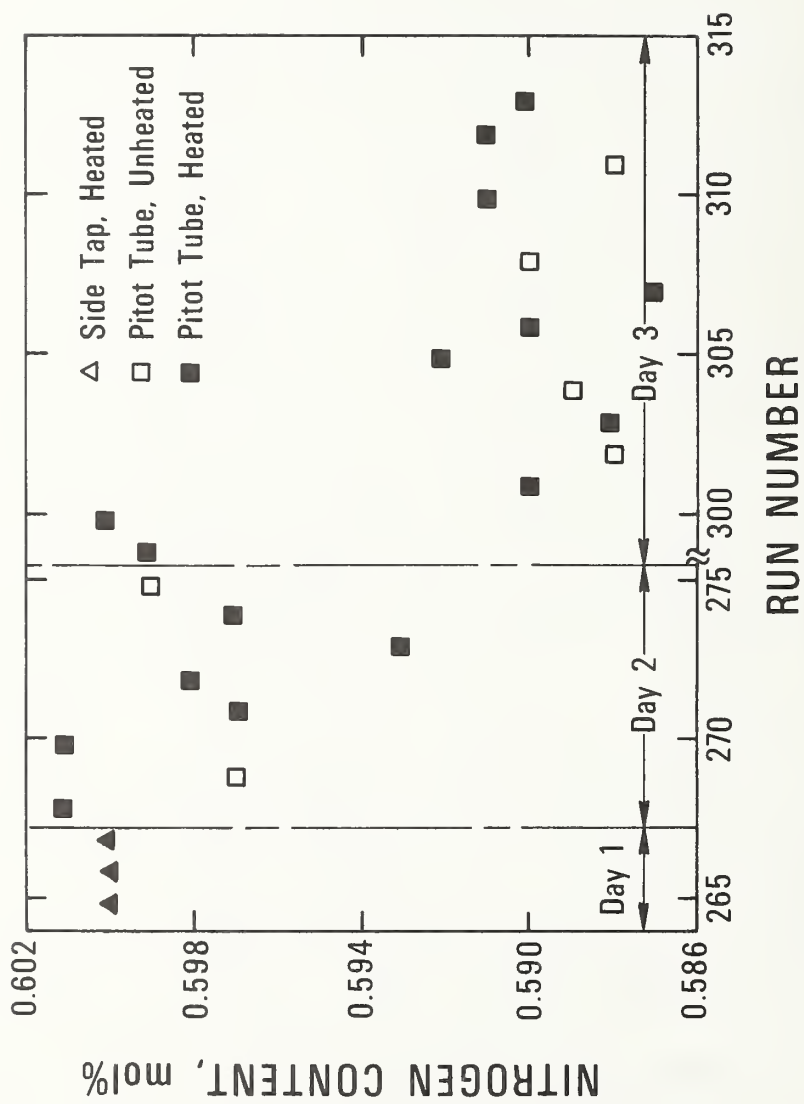


Figure 5.12. Nitrogen content for general test 3.

Table 5.10. Estimated standard deviations in the composition, heating value and liquid density for the pitot tube as a function of heat input into the probe line.

Heater Input Power, Watts	Number of Points	Estimated Standard Deviation in							Heating Value %	Liquid Density %
		Composition, mol %								
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀			
0	4	0.0006	0.042	0.011	0.024	0.003	0.006	0.06	0.04	
1.7	4	0.001	0.027	0.011	0.011	0.003	0.004	0.03	0.03	
6.7	4	0.002	0.044	0.033	0.013	0.002	0.001	0.04	0.03	

the vaporizer to be 1.0 psi (6.9 kPa) and 1.8 psi (12.4 kPa), respectively.) When controlling the sampling rate with the needle valve, the sampling rate had fluctuations of $\sim \pm 50$ percent of the average reading.

Table 5.11 and figure 5.13 show that the amount of pressure drop across the needle had no apparent effect on the sampling precision. However, there may have been sufficient resistance in the line to the vaporizer to prevent backflashing. This test was repeated in the second flow facility test.

Test 5

The first test in this phase of work clearly indicated that an accumulator was needed. However, the minimum acceptable residence time in the accumulator was not determined. In this short test we varied the residence time between 10 and 22 seconds. Residence times for past tests were in the range of 15 to 30 seconds. The 10 second residence time corresponded to a full scale reading on the rotameter used to measure the sampling rate. Figure 5.14 shows the heating values for this short test. Although the first point (293) appears too high it still is well within the error limits of gas analysis alone. There were too few data points to do a statistical analysis on each residence time. However, an analysis of the whole group indicated that the estimated standard deviation in computed heating values was comparable to the best values obtained in other laboratory tests (~ 0.02 percent).

Test 6

In all of the previous laboratory tests, prepared mixtures containing nitrogen and methane through butane were used. The LNG for the first flow facility test was prepared by condensing natural gas obtained from the local gas utility. The liquid contained roughly 0.2 mol percent hexanes and higher hydrocarbons. It was thought that the presence of heavy components may have caused some of the sampling problems experienced in the first flow facility test. Therefore, additional natural gas from the local utility was condensed and transferred into the laboratory apparatus. While holding all operating variables constant, a series of runs was made to determine the effect of heavy components on the sampling precision.

Table 5.12 lists the average values and estimated standard deviations for this test; figure 5.15 shows the deviation in the heating value. (The deviation is defined here as the difference between the measurement and average value divided by the average value for the given mixture.) Runs were made with different nitrogen levels. All of these analyses were made using column configuration A (see section 4.3.1.1). The estimated standard deviations for the first two days were comparable to previous laboratory tests. (The 0.005 mol percent value for the estimated standard deviation of nitrogen was typical for the gas chromatograph used on this test.) However, the imprecision was much greater in the heating value on the last day. This

Table 5.11. Estimated standard deviations in the composition, heating value and liquid density with high and low pressure drops across the needle valve upstream of the reference vaporizer.

Pressure Drop Across Needle Valve	Number of Points	Estimated Standard Deviation in							Heating Value %	Liquid Density %
		Composition, mol %								
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀			
High	14	0.001	0.024	0.014	0.014	0.001	0.005	0.005	0.03	0.03
Low	14	0.001	0.034	0.011	0.016	0.005	0.007	0.007	0.05	0.04

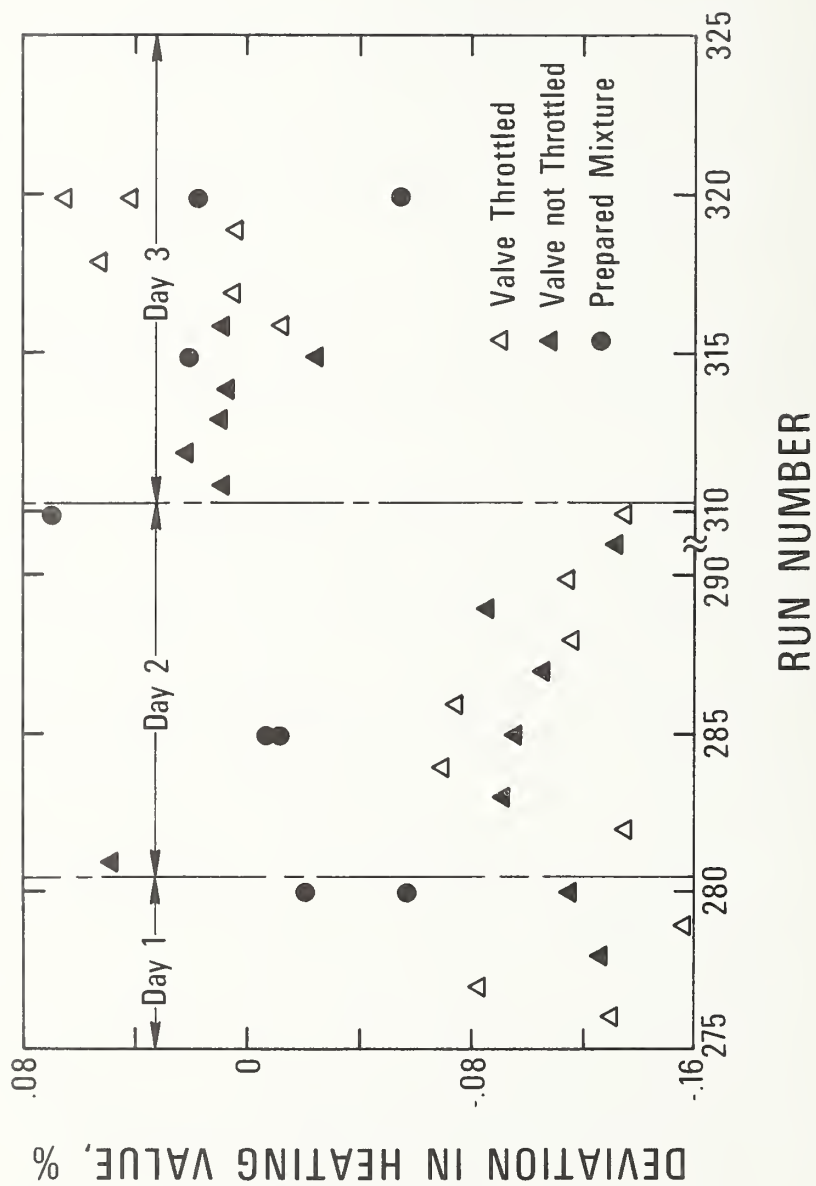


Figure 5.13. Deviations in the computed ideal gas heating values for general test 4.

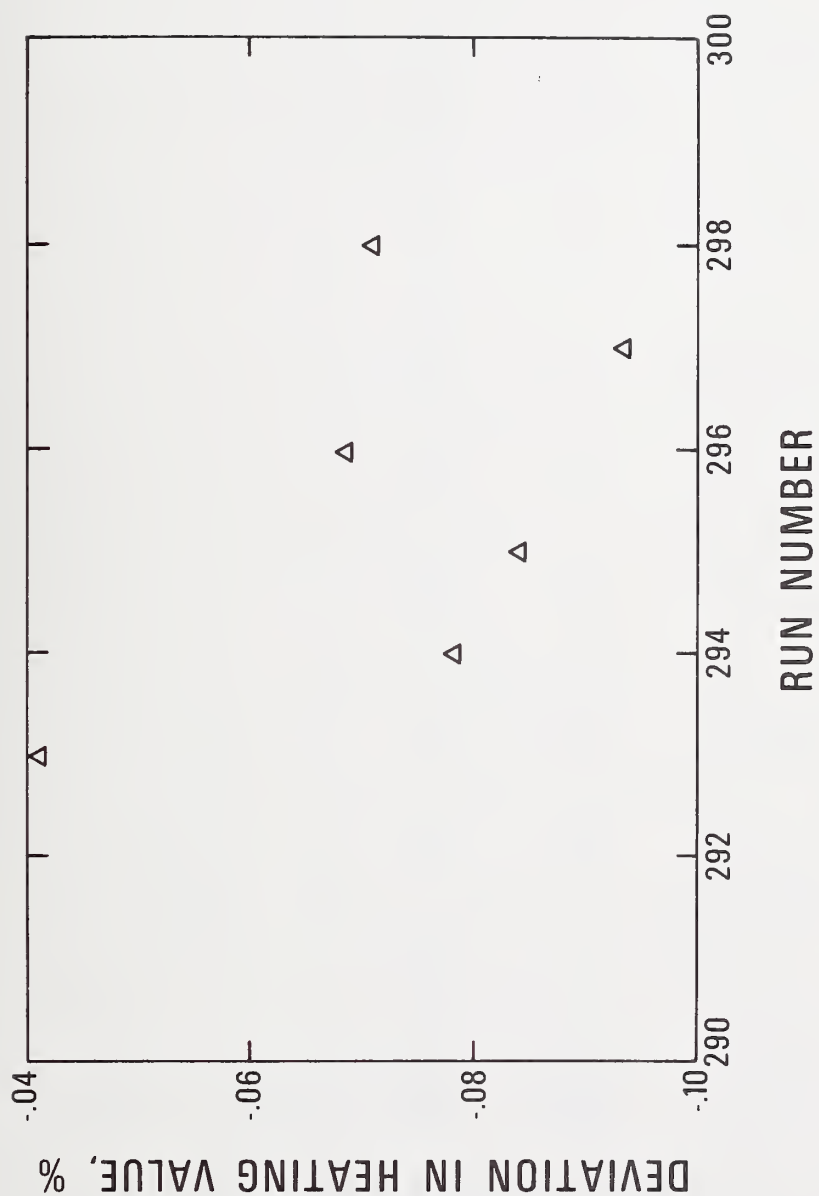


Figure 5.14. Deviations in the computed ideal gas heating value for general test 5. Residence times for this test are given in table 5.7.

Table 5.12. Average values and estimated standard deviations in composition, ideal gas heating value, and liquid density for LNG mixtures containing heavy components.

Date	Number of Points	Average Value and Estimated Standard Deviation in Composition, mol %											Heating Value BTU/SCF (MJ/m ³)	Liquid Density g/cm ³
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	C ₆ ⁺	CO ₂	O ₂		
5/11/77	6	0.132 0.005	84.307 0.024	10.839 0.008	3.403 0.015	0.325 0.002	0.740 0.004	0.124 0.001	0.112 0.001	0 0	0.007 0.003	0.012 0.006	1175.6 0.4 (43.791) (0.015)	0.4607 0.0001
5/12/77	8	0.716 0.005	83.850 0.013	10.789 0.008	3.338 0.005	0.322 0.0005	0.730 0.003	0.122 0.001	0.112 0.001	0.001 0.003	0.005 0.002	0.015 0.002	1167.9 0.2 (43.505) (0.007)	0.4646 0.00004
5/16/77	8	2.368 0.006	82.372 0.026	10.594 0.015	3.322 0.009	0.316 0.002	0.732 0.005	0.123 0.001	0.117 0.007	0.033 0.041	0.010 0.002	0.013 0.005	1150.8 1.4 (42.868) (0.052)	0.4707 0.0001

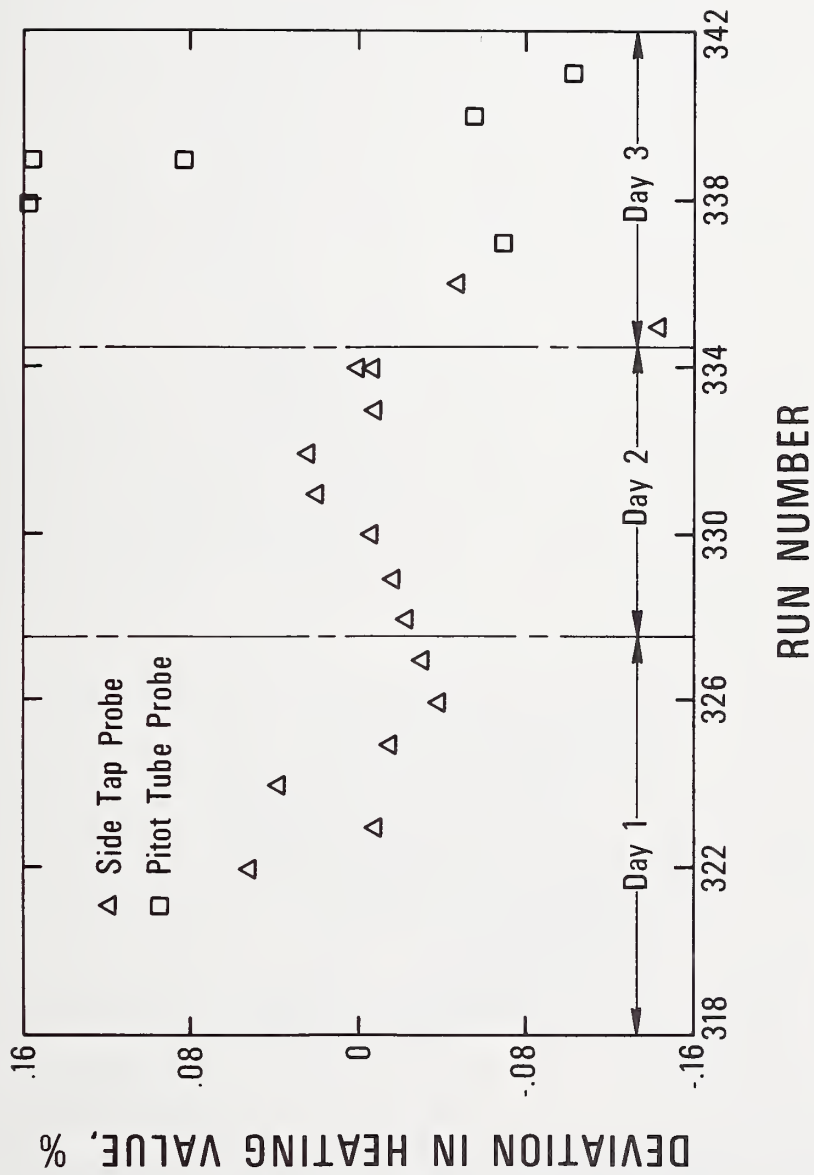


Figure 5.15. Deviations in the computed ideal gas heating values for general test 6.

was due to an increase in the scatter of the normal pentane and the C₆+ component peaks. An estimated standard deviation of 0.04 mol percent in the C₆+ peak corresponds to nearly 2 Btu/scf (0.08 MJ/m³) or 0.17 percent in the ideal gas heating value. The problem was not in sampling but in the inability of the digital integrator to correctly determine the area of the peak. (This problem is discussed in section 4.3.1.1).

At the time of these tests we did not have a calibration gas containing pentanes; therefore, response factors for the pentanes and C₆+ (assuming the peak contained equal parts of n-hexane and n-heptane) were estimated by taking the ratio of the response factor for the given component to that of n-butane [25] and multiplying that by the response factor for n-butane as determined from calibration. The same approach was used for the oxygen and carbon dioxide peaks except that nitrogen, instead of n-butane, was used in the ratio.

Based on the results of this test, the presence of heavy components does not affect the sampling precision but they can affect the analytical precision. This test pointed out the analytical problem of correctly integrating the C₆+ backflush peak.

5.2. Confirmation Tests

It was important that the conclusions obtained from the small scale laboratory tests be confirmed in a facility comparable to the proposed industrial installation. Three confirmation tests were made -- two at the NBS LNG Flow Facility and one aboard the LNG tanker El Paso Consolidated. The shipboard test was made after completing the laboratory tests and between the two flow facility tests.

5.2.1. NBS LNG Flow Facility Test 1

The LNG flow facility [26] is an outdoor pilot plant which has been used primarily for LNG flowmeter measurements. Figure 5.16 shows a schematic of the facility and table 5.13 lists the pertinent operating data. For the sampling tests a 3 ft (0.9 m) long test section was inserted roughly 10 ft (3 m) downstream of the recirculation pump. All lines in the flow loop were 2.5 in (72 mm) diameter stainless steel pipe and all lines except the test section were vacuum jacketed.

Table 5.13. LNG flow facility operating characteristics

Temperature Range	95-140 K
Temperature Stability	± 1 K or better
Pressure Range	19-110 psia (0.13-0.76 MPa)
Pump Discharge Pressure (above weigh tank pressure)	2.7-30 psid (19-207 kPa)
LNG Flow Rate	20-200 gpm (76-760 Lpm)
Reynold's Number	10,000-100,000

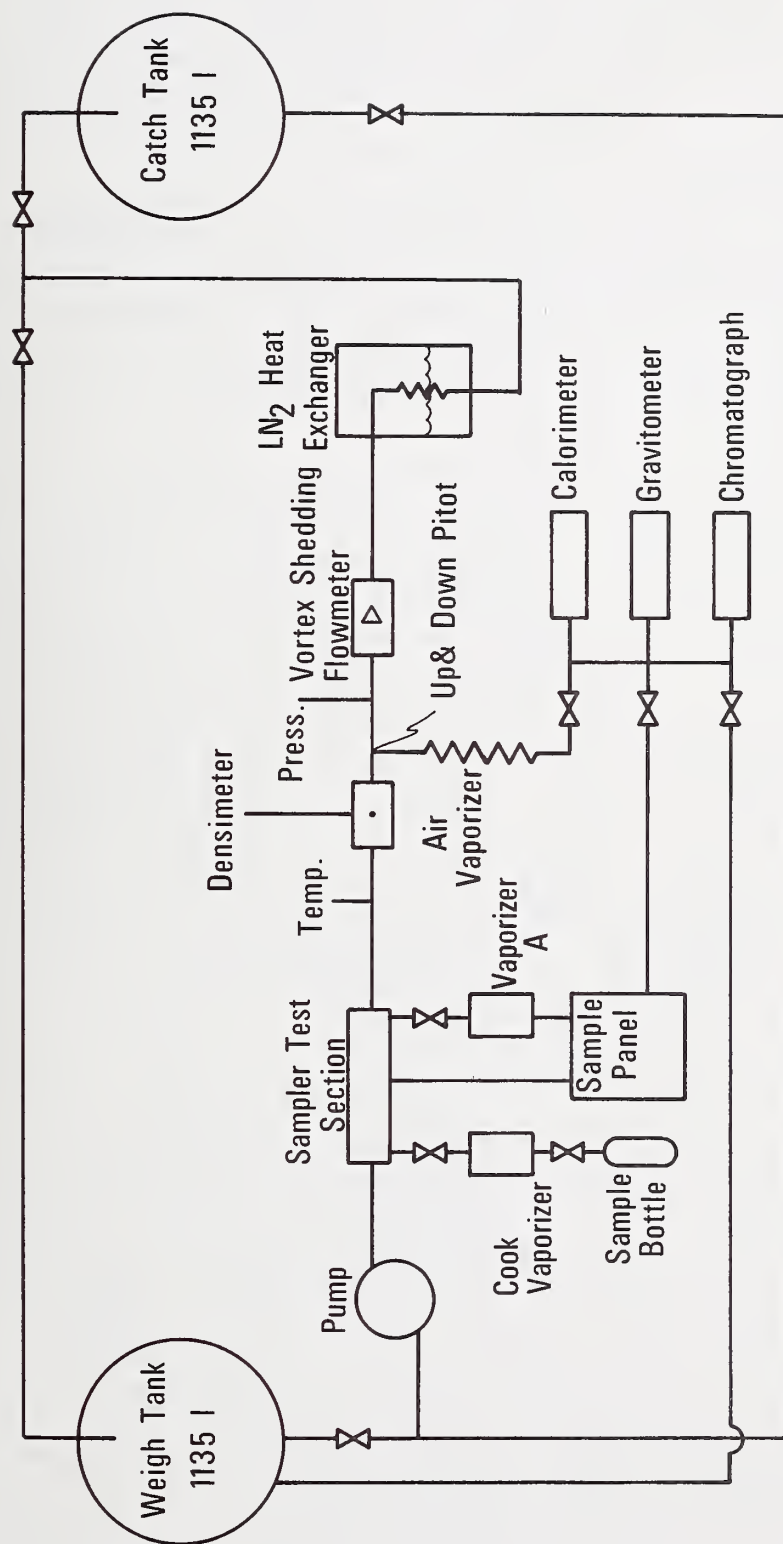


Figure 5.16. Flow schematic for the first LNG flow facility test.

Vaporized samples from the test section were collected in 150 cm³ sample cylinders for analysis in the laboratory GC. For these tests we used column configuration A (see section 4.3.1.1). The vaporized samples also could be sent to a gas gravitometer and combustion calorimeter. These units were located approximately 50 ft (15 m) away in a constant temperature room. The residence time of gas in the line between the sample panel and calorimeter was 1.6 minutes. Additional gas samples could be taken just upstream of the calorimeter and gravitometer.

It was possible to send gas samples directly to a process gas chromatograph. But since it measured only the concentration of nitrogen, methane, ethane, carbon dioxide and propane it was not used in these tests. Also, there was a pitot tube (up and downstream facing pitot)-air vaporizer sampling system [27] downstream of the sampling test section. It was used only in the first test.

During the initial startup the flow loop was purged with dry nitrogen and then cooled down with liquid nitrogen which was recirculated through the flow loop. After cooldown, LNG was transferred into the weigh tank. For the sampling tests the LNG was pumped from the bottom of the weigh tank and returned to the top of the weigh tank. The temperature was controlled by adjusting the liquid level in the liquid nitrogen heat exchanger.

The "true" composition of the LNG in the flow facility could not be measured. This limited the tests to comparing only the precision of the various sampling systems. However, the composition could be held constant throughout an operating day. System venting during shutdown caused day to day shifts in the composition. LNG for this test was made by condensing the natural gas supplied by the local gas utility. The gas was dried with a desiccant and the carbon dioxide was removed by filtering the liquid [27].

In Test 1 we wanted to compare three probes -- a side tap, a side tap with a capillary opening and an upstream facing pitot tube; two vaporizers -- a water heated coil and vaporizer A; and the Cook sampler. Figure 5.17 shows a schematic of the sampling system (a sample accumulator was not used). All three probes were mounted horizontally in the test section and were 6 in (152 mm) apart. The test section was enclosed in a 5 in (127 mm) section of copper tubing. The annular space was evacuated to provide insulation. (However, during the tests it became obvious that the vacuum was very poor because a heavy layer of frost formed on the outer tube.) All three probes were made of 1/8 in (3.2 mm) O.D. by 0.101 in (2.56 mm) I.D. stainless steel tubing. The probe lines were vacuum jacketed 6-1/2 in (165 mm) beyond the 5 in (127 mm) tube. The pitot tube had a 90° bend with the intake on the flow section center line.

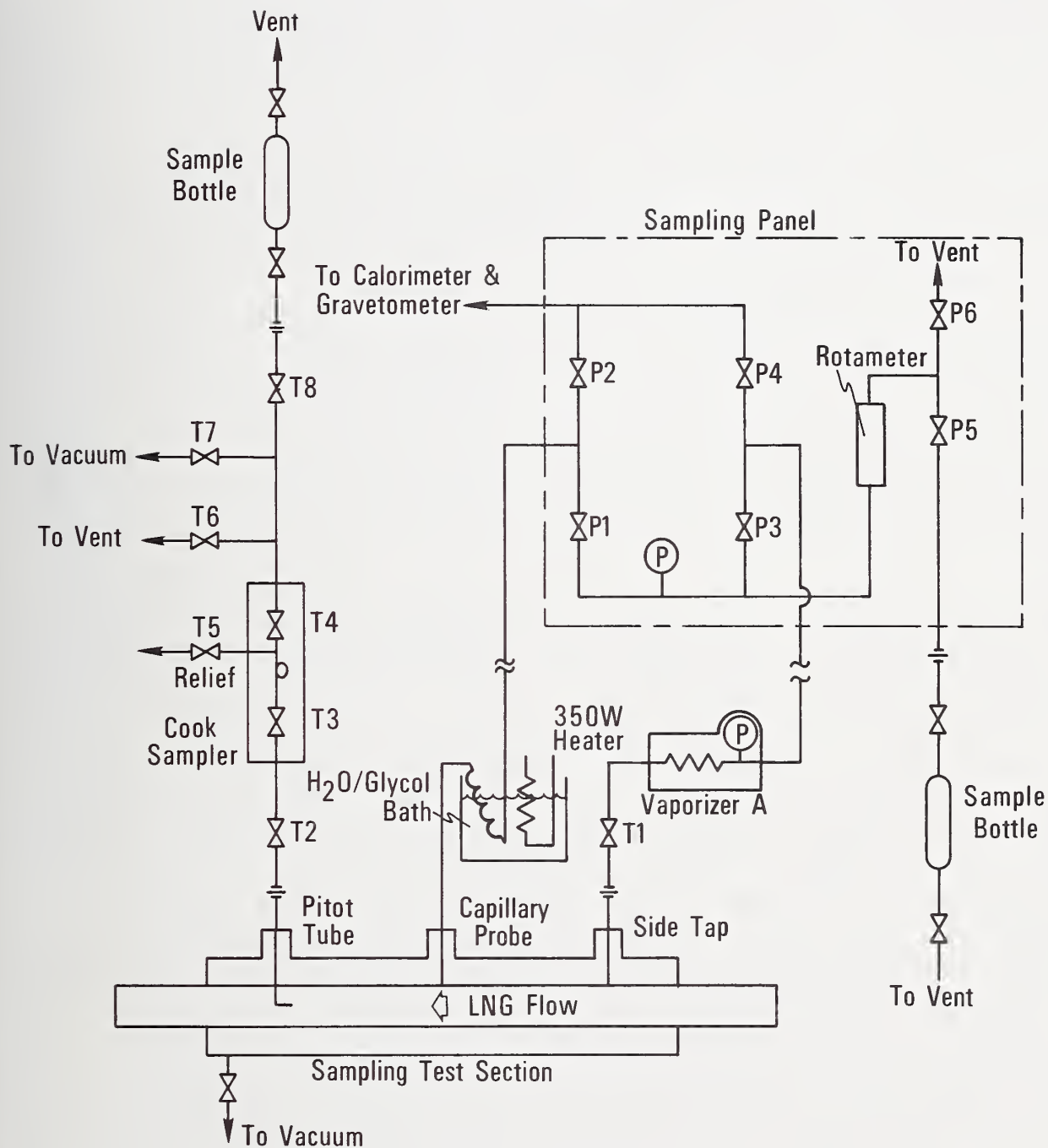


Figure 5.17. Schematic of sampling system used in flow facility test 1.

It was thought that if part of the pressure drop between the probe and vaporizer could be taken in the liquid phase there would be no danger of backflash; hence, it would not matter how the sample was vaporized downstream. Therefore, a capillary probe was built which contained a 1/4 in (6.4 mm) long section of 0.007 in (0.18 mm) I.D. tubing. This was soldered into the 1/8" (3.2 mm) O.D. tubing. The capillary section produced roughly 5-1/2 psi (38 kPa) pressure drop for a liquid flow corresponding to 8 slpm of gas flow.

The water heated vaporizer consisted of 10 ft (3 m) of 1/4 in (6.35 mm) O.D. x 0.190 in (4.83) I.D. copper tubing immersed in a water-glycol bath which was at $\sim 30^{\circ}\text{F}$ (-1°C). During operation the coil would have a ice layer which extended about 3 ft (0.9 m) along the tubing in the bath.

5.2.1.1. Test results

The results of this test were extremely poor; the best agreement between two samples was 0.04% in the heating value. However, this was fortuitous because the typical scatter was on the order of one percent. The scatter was unaffected by the probe and vaporizer used. It was found that the heating values of the samples taken five minutes apart from the same sampling system varied by 0.7 percent; samples taken simultaneously from the same stream at the panel and at the gravitometer inlet differed by 1.5 percent. However, during all of these tests the calorimeter and gravitometer remained steady. (The gas specific gravity is much less sensitive to composition than the heating value, e.g., in the composition range studied a 1.5 percent change in the heating value corresponds to only 0.4 percent change in the specific gravity.)

Initially, it was thought that the scatter could be caused by the different methods of filling the sample cylinder. Samples taken at the panel were purged and filled 14 times; the sample cylinders at the gravitometer/calorimeter inlet were filled by continuous purge through. To test the effect of sample cylinder filling procedure on precision, one of the calibration gases was connected to the sample panel. Using comparable sample flows and pressures to those used in the other tests, samples were taken at both the panel and the gravitometer/calorimeter inlet. The resulting sample compositions were within the error of the gas analysis alone; therefore, the sample cylinder filling procedure was not a problem.

The possibility that the scatter was caused by either liquid composition fluctuations or by the presence of vapor in the test section was considered. The stability of the densimeter in the LNG flow facility indicated that there were no composition fluctuations. The possibility of two phase flow was eliminated because the vortex shedding flowmeter would have behaved erratically in two phase flow. At this point it was decided to make additional laboratory tests (see section 5.1.3.3) to determine the cause of the poor sampling precision.

Even with the erratic sampling precision, the following qualitative results were obtained:

1. It was not possible to obtain reliable samples from the Cook sampler as tested; this sampler should have been insensitive to the problems experienced with the continuous samplers. However, it is possible that valve T2 (figure 5.17) was leaking during the warm up period or that the reduction in sample pressure when filling the sample cylinder caused partial condensation. (The ambient air during these tests was 30 to 40°F (-1.1 to 4.4°C)).

2. Based on the band width of the gravitometer trace, the up and down pitot-air vaporizer and the capillary probe-water vaporizer systems produced more precise samples than did the side tap-vaporizer A system. The gravitometer band width for the capillary probe-water vaporizer samples and gravitometer calibration gas (air) were comparable. Also, it was found that the heating values for the side tap-vaporizer A samples were 0.5 to 1 percent higher than those from the other two systems; the heating value difference between these two systems was less than 0.2 percent.

3. The capillary probe is not a feasible probe in industrial applications where entrained solids could cause plugging. In the flow facility there were small amounts of solid carbon dioxide which plugged the capillary line.

5.2.2. Shipboard Tests

Using the information obtained in the laboratory and first flow facility tests, a full scale sampling system was designed for tests aboard the LNG tanker El Paso Consolidated. Sampling tests were made at Canvey Island, England during the ship's cargo system trials. Two sampling systems were installed -- a liquid sampling system was installed on the 3 inch (76 mm) diameter header to the ship's LNG vaporizer; a gas sampling system was placed on the 16 inch (406 mm) diameter vaporizer outlet header. Sampling the gas phase provided a check on the liquid sampling system.

Figure 5.18 shows a schematic of the liquid sampling system and table 5.14 lists the component dimensions. The system had a 1/4 inch, schedule 80 pipe (13.7 mm O.D. x 7.67 mm I.C.) side tap probe mounted horizontally on the header. All valves, fittings and tubing upstream of the sample panel were stainless steel; tubing in the panel was copper and the other components were brass. Except for the filter connections and union just upstream of the vaporizer, all connections upstream of the vaporizer were socket welds. The line between the header and vaporizer was wrapped with a one inch (25 mm) layer of fiberglass pipe insulation and sealed with duct tape. Valves T1 and T2 were bellows valves with the valve components welded together. (We have had success using these valves in cryogenic applications.) Valve T2 was a metering valve; it and the back pressure regulator were used to control the sampling rate.

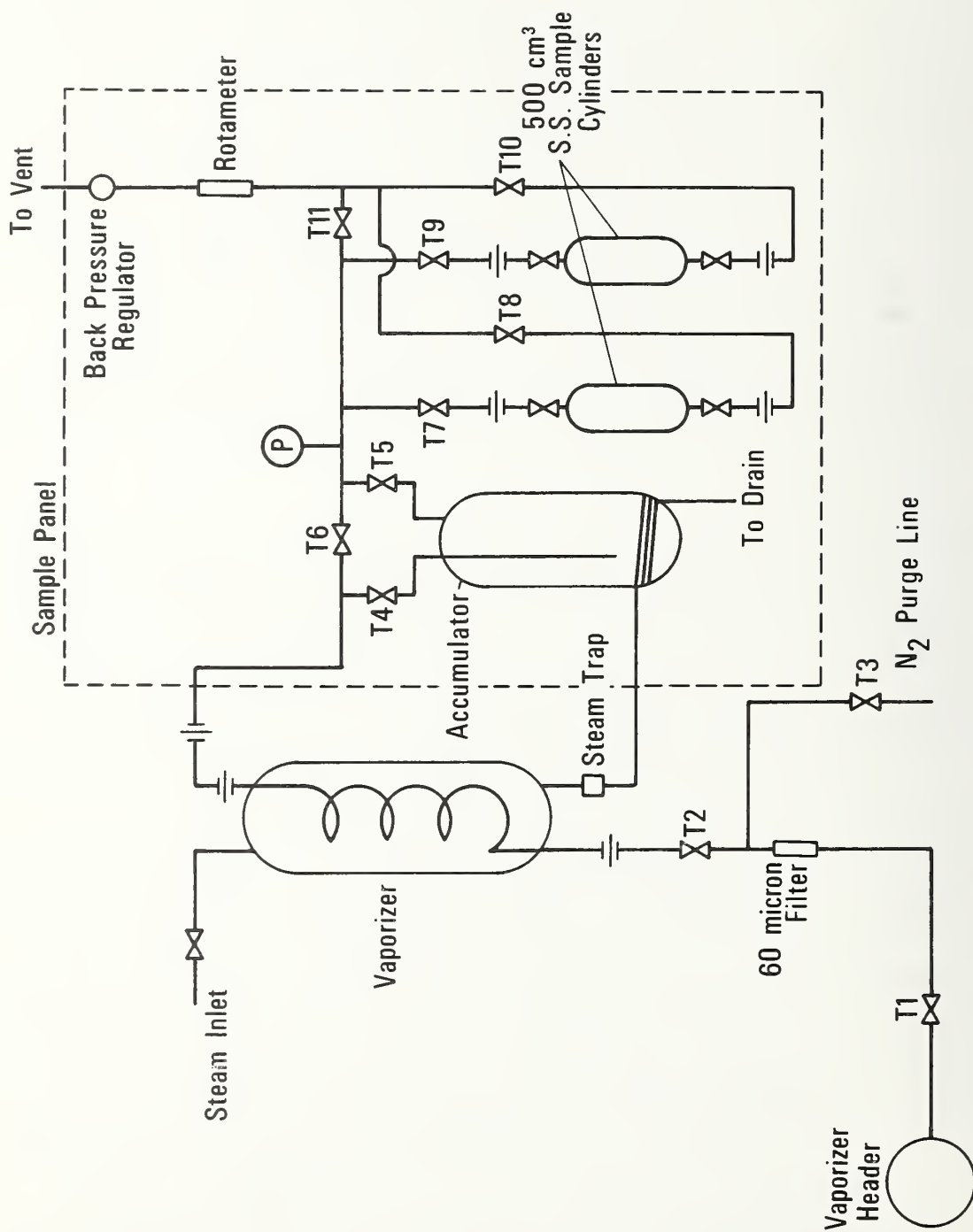


Figure 5.18. Schematic of liquid sampling system used in shipboard tests.

Table 5.14. Dimensions of LNG Sampling System used on Shipboard Tests

Component	Length inches (mm)	Outer Diameter inches (mm)	Inner Diameter inches (mm)
Vaporizer Header	--	3.5 (88.9) (3 in pipe)	
Side tap and line between header and Valve T1	4½ (114)	0.540 (13.7) (½ in Sch 80 pipe)	0.302 (7.67)
Line between Valve T1 and filter (con- tained a long radius 90° bend)	11 (279)	0.25 (6.4)	0.180 (4.57)
Line between filter and Valve T2	~3 (76)	0.25 (6.4)	0.180 (4.57)
Line between Valve T2 and union at vaporizer	18 (460)	0.125 (3.18)	0.101 (2.56)
Vaporizer tubing (formed into a 4 in (102 mm) diam- eter helix)	45 (1140)	0.188 (4.76)	0.164 (4.15)
Vaporizer Casing (with welded cap ends)	20 (508)	6.00 (150)	5.94 (151)
Line from Vaporizer to union (contained a long radius 90° bend)	11 (279)	0.25 (6.4)	0.180 (4.57)
Line to Valve T3	14 (356)	0.25 (6.4)	0.180 (4.57)
Accumulator (with welded cap ends)	24 (610)	6.00 (152)	5.94 (151)

The nitrogen purge connection (which was insulated up to valve T3) was used for displacing air from the sampling system; it was located downstream of the 60 micron filter so that nitrogen could be used to back purge the filter, if necessary. However, the filter never clogged during these tests.

Because of film boiling, a precise (± 20 percent) design of the sample vaporizer would have been extremely tedious. Therefore, the design was based on the warming of methane gas from 115 to 310 K. A flow rate of 65 slpm was used. We assumed that the steam side temperature was constant at 340°F (171°C) this corresponds to the temperature of 205 psia (1.41 MPa) steam isentropically expanded to 60 psia (0.4 MPa). The heat transfer resistances on the steam side and in the tube wall were negligible compared to the gas phase resistance; properties of methane [28,29] at 150 K were used in the calculations. The final vaporizer tube length was twice the length computed using the above assumptions.

The steam inlet line, vaporizer and condensate line were insulated; this was done primarily for personnel safety. Condensate from the vaporizer was used to warm the bottom of the accumulator to provide additional mixing.

The accumulator was sized to provide a 30 second residence time for a sample rate of 65 slpm and sample pressure of 50 psig (0.45 MPa). The length to diameter ratio was 2.5 and the volume was 11 liters.

The sample panel layout was designed so that the sample flow could be maintained with or without sample cylinders in the system. The back pressure regulator was extremely useful for making minor changes in the sampling rate and setting the sample cylinder pressure.

The gas sampling system consisted of a 1/4 inch, schedule 80 pipe (13.7 mm O.D. x 7.67 mm I.D.) side tap mounted vertically on the top of the outlet header, a block valve and 1/4 inch (6.35 mm) O.D. by 0.190 inch (4.83 mm) I.D. copper tubing going to the sample panel. This panel had the same layout as the liquid sample panel but included a needle valve upstream of the accumulator for flow control. This system was designed for a vaporizer outlet pressure comparable to the inlet pressure (65 psig (0.055 MPa)). However, the outlet pressure was only 1.5 to 2 psig (0.112 to 0.115 MPa); this reduced the sampling rate to approximately 1 slpm from the anticipated 10 or more slpm.

Gas analyses were made using a gas chromatograph containing a 30 ft (9.1 m) long column of DC 200/500 on Chromasorb P and a 5 ft (1.5 m) molecular sieve 5A (MS) column. The column valve configuration permitted backflushing the DC 200/500 column and bypassing the MS column. Nitrogen and methane were resolved on the MS column. Ethane through the butanes were separated on the DC 200/500 column; the column was backflushed to obtain a C₅⁺ peak. Digital integrator A was used to measure peak areas. The precision of the analytical system was found to be 0.06 percent in the computed heating value.

5.2.2.1. Test Results

The primary purpose of this test was to examine the precision of the LNG sampling system. The LNG was totally vaporized downstream of the sampling probe and the analysis of this gas was to serve as the basis on which to evaluate the liquid sample analysis. However, the analyses of the vaporized gas produced erratic results that are felt to be more a function of the low pressure of the gas than composition change in the pipeline. Of the six sets of sampling data taken, only the second set contained sufficient samples taken under constant vaporizer conditions to directly compare the gas and liquid sample compositions. See figure 5.19. Since composition variations were probably less of a factor than the gas analyses, separate means and standard deviations were computed for the liquid samples and for the gas samples for each of the six data sets. See table 5.15. The heating values are computed using the A.G.A. method [1] and assumes equal parts of iso and normal pentane; the liquid phase densities were computed from the extended corresponding states model [3] (Appendix 7.5 lists the compositions and operating parameters for all of the tests).

During the first data set the vaporizer inlet pressure varied erratically (30-56 psig). Therefore, these samples were taken only to gain experience in operating the sampling system. For data set 6, the chromatograph-integrator system was behaving erratically with respect to the butane peaks. For these reasons, the results for these two data sets, as given in table 5.15, do not represent a properly operating system under stable operating conditions and were not included in subsequent data analysis.

Although the conditions under which liquid samples were taken for data sets 2, 3, 4, and 5 are considered acceptable for sampling purposes, some points were deleted from the data analyses. It was found that the LNG sampling system worked well at sampling rates of 12.5 slpm or greater; however, two samples, run No. 19 at 3.4 slpm and run No. 57 at 6.2 slpm, yielded poor results and were omitted from the analyses. Also during these tests the accumulator residence time of the "good" liquid samples ranged between 28 and 76 seconds, and on this basis run No. 25 was also omitted. Run No. 31 was omitted from the data analysis because it did not go through the accumulator and run No. 35 was omitted as the evidence indicated that it was an outlier. For data set 4, run No. 51 was analyzed three times; the first analysis, being completely wrong, was omitted.

An estimate of standard deviation was calculated based on data sets 2, 3, 4, and 5. For the liquid samples the value obtained was 0.72 Btu/scf with 17 degrees of freedom. An upper 99% confidence bound for the standard deviation based on this estimate is 1.18 Btu/scf, or 0.1% based on a heating value of 1200 Btu/scf.

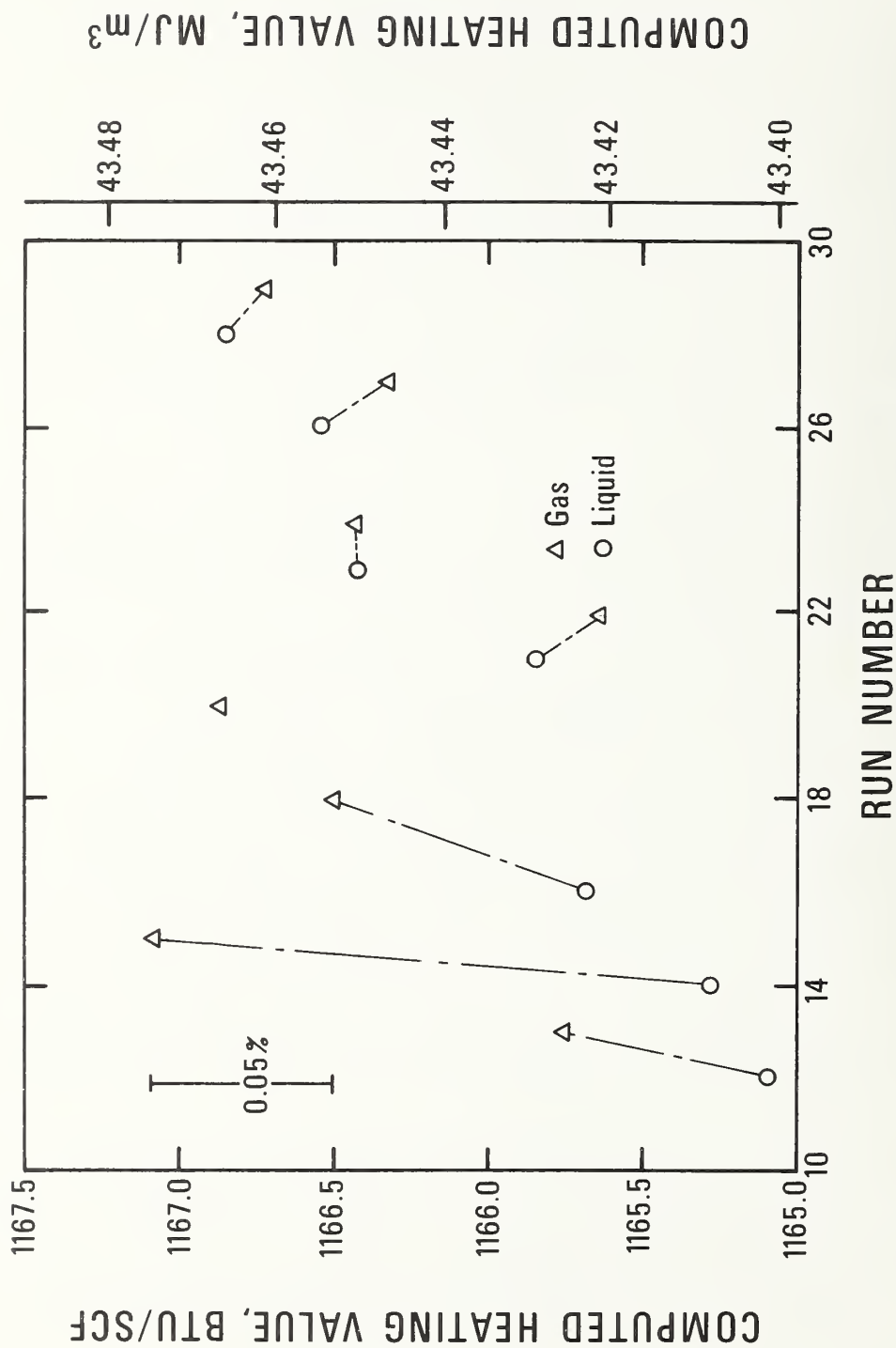


Figure 5.19. Computed real gas heating values of shipboard data set 2 samples.

Table 5.15. Average values and estimated standard deviations in composition, real gas heating value, and liquid density for shipboard tests.

Data Set	Sample	Number of Points	Average Value and Estimated Standard Deviation in								Heating Value BTU/SCF (MJ/m ³)	Liquid Density g/cm ³
			Composition, mol %									
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	i C ₄ H ₁₀	n C ₄ H ₁₀	C ₅ ⁺			
1 * T=114.6	L	7	0.201 0.143	85.864 3.101	10.063 2.252	2.983 0.716	0.452 0.155	0.416 0.134	0.020 0.020	1165.8 35.9 (43.428) (1.338)	0.4682 0.0107	
	G	1	0.209	83.762	11.553	3.442	0.541	0.494	0.0	1176.7 (43.833)	0.4752	
2 T=114.6	L	7	0.110 0.003	85.149 0.036	10.558 0.024	3.176 0.013	0.511 0.004	0.460 0.003	0.045 0.003	1165.8 0.5 (43.428) (0.020)	0.4712 0.0001	
	G	8	0.109 0.007	85.112 0.049	10.573 0.051	3.184 0.009	0.515 0.005	0.460 0.002	0.047 0.057	1166.3 0.5 (43.446) (0.020)	0.4713 0.0001	
3 T=114.6	L	4	0.015 0.017	82.544 0.031	12.353 0.038	3.863 0.023	0.623 0.005	0.563 0.004	0.040 0.028	1195.8 0.41 (44.547) (.015)	0.4799 0.0001	
	G	4	0.016 0.016	82.258 0.563	12.611 0.496	3.898 0.101	0.614 0.027	0.553 0.002	0.051 0.008	1198.3 5.6 (44.638) (0.211)	0.4806 0.0016	
4 T=115.0	L	8	0.0006 0.0015	79.045 0.080	14.515 0.050	4.854 0.023	0.800 0.005	0.719 0.004	0.067 0.003	1236.2 0.9 (46.050) (.035)	0.4914 0.0009	
	G	4	0.001 0.002	78.971 0.134	14.530 0.075	4.868 0.024	0.802 0.008	0.721 0.004	0.108 0.082	1237.9 2.8 (46.112) (0.102)	0.4916 0.0007	
5 T=116.0	L	2	0.0	78.367 0.055	15.042 0.046	5.000 0.013	0.808 0.0015	0.720 0.006	0.064 0.010	1242.6 0.7 (46.289) (.027)	0.4917 0.0002	
	G	2	0.001 0.002	78.280 0.091	15.139 0.037	5.006 0.064	0.800 0.009	0.714 0.002	0.062 0.006	1243.0 0.9 (46.304) (0.035)	0.4919 0.0003	
6 * T=116.5	L	7	0.0	77.905 0.09	15.361 0.07	5.112 0.022	0.821 0.006	0.733 0.005	0.078 0.007	1247.5 0.9 (46.471) (.035)	0.4926 0.0006	
	G	4	0.0	78.216 0.356	15.229 0.284	5.081 0.106	0.692 0.225	0.724 0.004	0.057 0.040	1242.6 5.6 (46.287) (0.209)	0.4912 0.0015	

*These tests occurred during unstable conditions (refer to text for details) and the values for the means and standard deviations are not meaningful.

A commercial "primary standard" calibration gas was used for gas chromatograph calibrations. During the calibrations it became obvious that the reported and actual calibration gas compositions were different. This discrepancy was found by comparing the nitrogen to methane response factor ratio with the ratio obtained from Dietz's data [25]. Table 5.16 gives the manufacturer's stated composition and error limits along with the composition determined in our laboratory. Column configuration A was used with mixture H as the calibration gas. The difference between the stated and actual composition represents a 0.5 percent error in the heating value. This means that if the stated compositions had been used there would have been a bias of 0.5 percent in the computed heating value. This experience points out the importance of accurately knowing the composition of calibration gases used in custody transfer. If possible the stated composition should be verified by an independent gas analysis laboratory.

5.2.3 LNG Flow Facility Test 2

At this point, we had a full scale LNG sampling system which had worked in the field. However, another field test was necessary to determine:

- a) the criteria for establishing sampling rate,
- b) the effect of using steam versus electricity for sample vaporization,
- c) the effect of not using a needle valve to control sampling rate, and
- d) the effect of heat leak.

Figure 5.20 shows the flow schematic for this test. We built three vaporizers -- two steam heated and one electrically heated. One of the steam vaporizers had the same dimensions as the electric vaporizer; the second steam vaporizer had a 50 percent larger tube inner diameter and the tube length was 40 percent longer than the other two vaporizers. Table 5.17 gives the important dimensions of the vaporizers and sampling systems; figure 5.21 is a schematic of one of the sampling systems. The old test section was replaced with a test section containing two side taps mounted horizontally. The side taps were 1/4 inch schedule 80 (13.7 mm O.D. x 7.67 mm I.D.) stainless steel pipe. Except for the vaporizers, all dimensions on both sampling systems were identical. Therefore, by operating two vaporizers simultaneously we made direct comparisons between vaporizer designs. The LNG sample lines to the vaporizers were wrapped with a 1/2 inch (13 mm) thick layer of fiberglass pipe insulation and sealed with duct tape. Also, the steam vaporizers, steam inlet lines and condensate lines were insulated for personnel safety.

Valve S1 was a block valve (see figure 5.21) and valve S2 a metering valve; both valves were all welded stainless steel bellows valves. Neither valve was degreased before use and stem extensions were not used.

Table 5.16. Reported and measured composition of commercially prepared primary standard calibration gas.

	Composition, mol percent						
	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	nC ₅ H ₁₀
Reported Values	0.545 ±0.0055	87.112 ±0.056	8.165 ±0.020	2.752 ±0.020	0.4495 ±0.0045	0.5570 ±0.0055	0.01999 ±0.0002
Measured Values	0.632 ±0.003 ¹	87.172 ±0.046	8.862 ±0.008	2.589 ±0.035	0.351 ±0.002	0.383 ±0.003	0.007 ±0.003

¹The interval given represents a 99% confidence interval and is based on 4 gas calibrations and 5 gas analyses of the mixture. In computing the confidence interval we assumed that there was no composition error in the calibration gas used to calibrate the GC and that the standard deviations of both calibrations and analyses were equal.

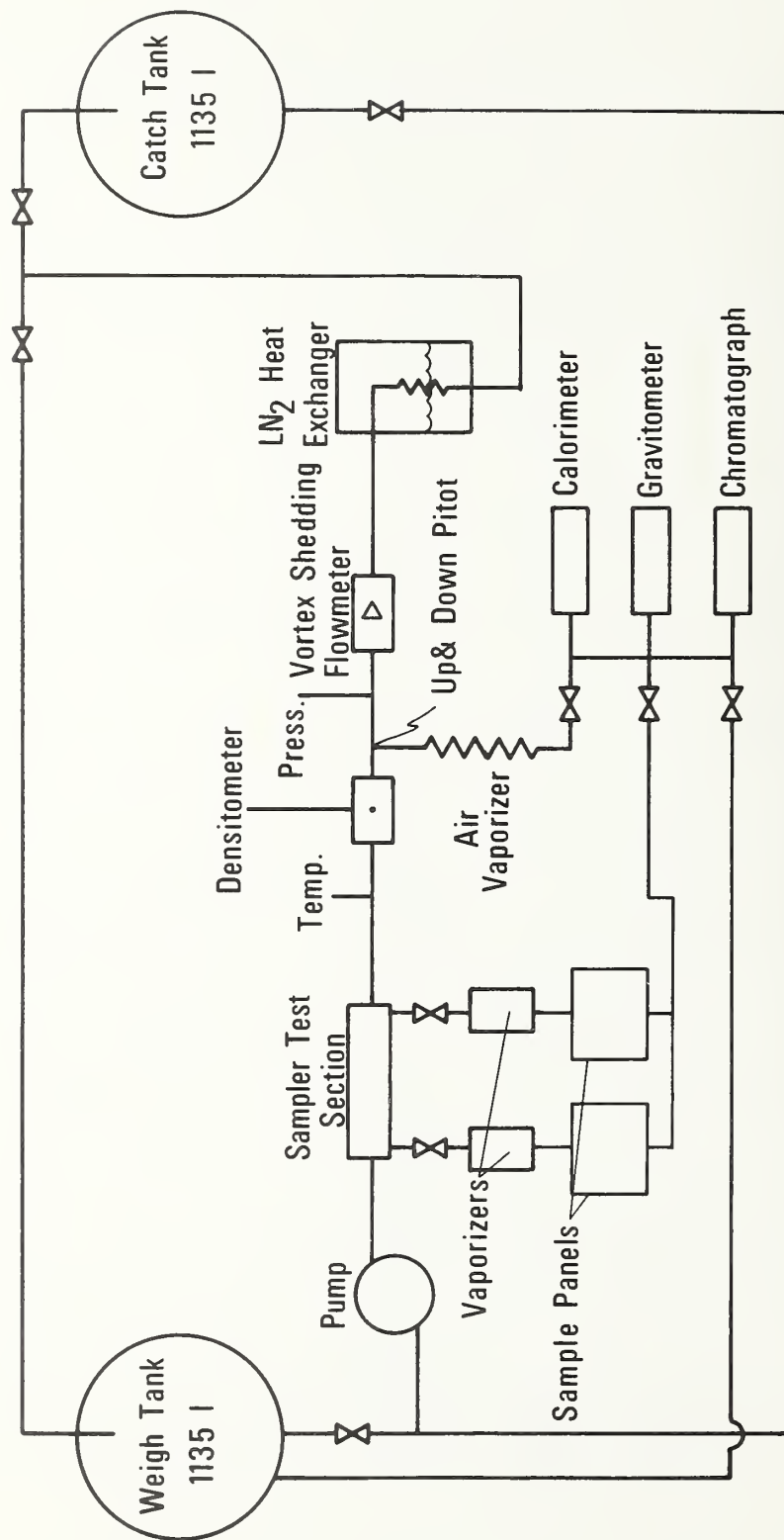


Figure 5.20. Flow schematic for the second LNG flow facility test.

Table 5.17. Dimensions of LNG sampling system used in second flow facility tests (see figure 5.21 for schematic of sampling system).

Component	Length Inches (mm)	Outer Diameter Inches (mm)	Inner Diameter Inches (mm)
Test section	36 (914)	3-1/8 (79.4)	2.812 (71.42)
Side tap and line between test section and valve S1 ¹	4-1/2 (114)	0.540 (13.7) (1/4 in. Sch 80 pipe)	0.302 (7.67)
Line between valve S1 and filter ¹ (contained a short radius 90° bend)	11 (279)	0.25 (6.4)	0.180 (4.57)
Line between filter and valve S2 ¹	2 (51)	0.25 (6.4)	0.180 (4.57)
Line between valve S2 and union at vaporizer ¹	13-1/2 (343)	0.25 (6.4)	0.180 (4.57)
Vaporizer 1 tubing	36 (914)	1/8 (3.18)	0.101 (2.56)
Vaporizer 2 tubing	50 (1270)	3/16 (4.76)	0.156 (3.96)
Vaporizer 3 tubing	36 (914)	1/8 (3.18)	0.101 (2.56)
For vaporizers 1 and 2 steam casing (with flat welded ends)	10 (254)	6 (152)	5.5 (140)
Impingement chamber (with flat welded ends)	1-1/2 (38)	1-1/2 (38)	1.376 (34.95)
Line between impinge- ment chamber and down- stream union	8 (200)	0.25 (6.4)	0.180 (4.57)
Accumulator ¹	16 (406)	5.125 (130.2)	4.805 (122)

¹ These dimensions apply to both sampling systems.

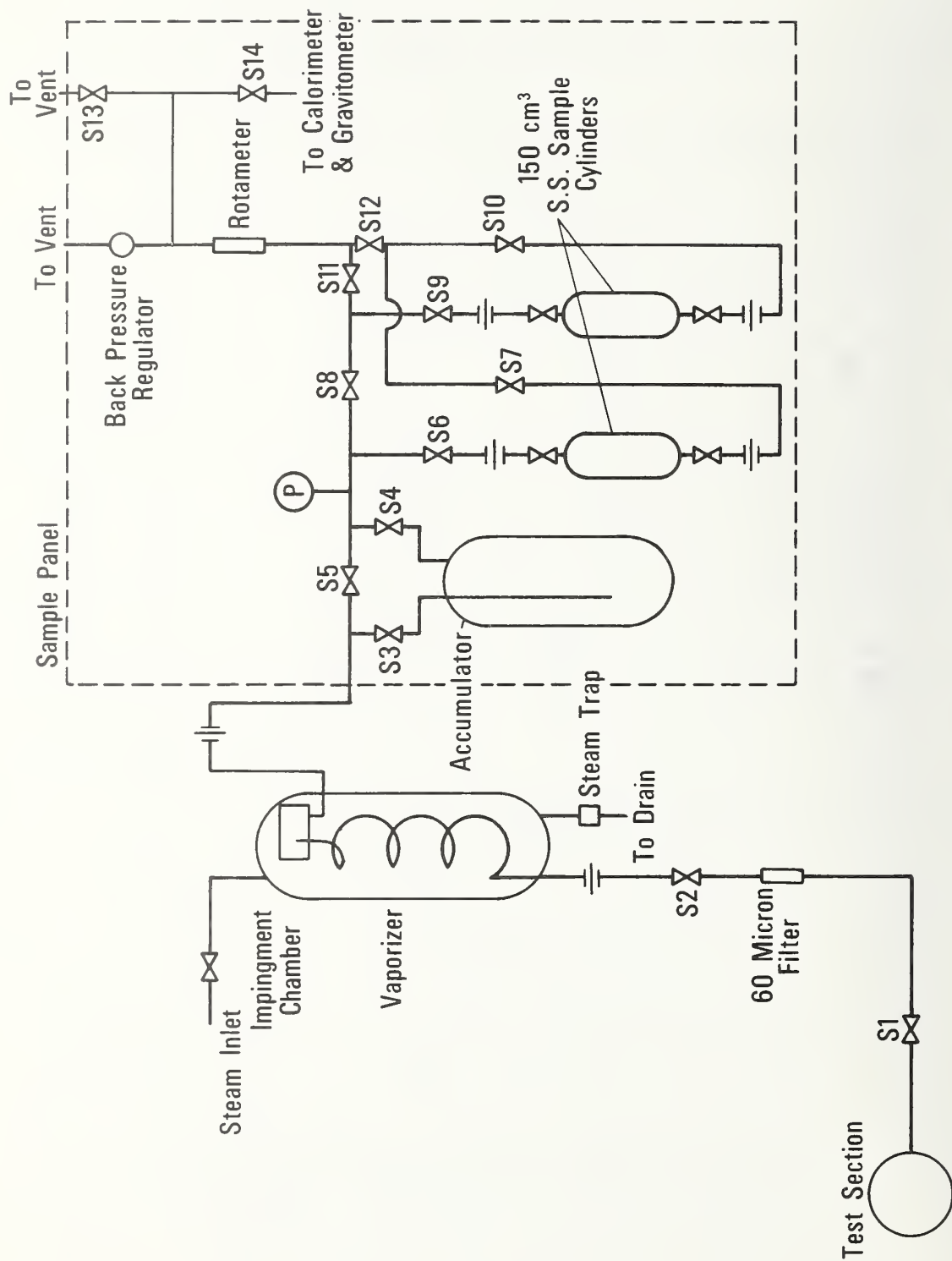


Figure 5.21. Schematic of liquid sampling system used in flow facility test 2.

The two steam vaporizer tube lengths were twice the tube length computed by considering only the heat transfer resistance of a pure methane gas phase (see section 5.2.2 for more details). For this test we used a steam generator which produced roughly 50 psia (0.34 MPa) saturated steam.

To prevent the entrainment of fine droplets in the steam vaporizer outlet steam, an impingement chamber was included in each steam vaporizer. The stainless steel chamber consisted of a 1-1/2 inch (38 mm) diameter cylinder 1-1/2 inches (38 mm) long. It was constructed so that the vapor plus mist leaving the vaporizer tube contacted the upper surface of the chamber. The combination of impingement, sudden flow decrease and flow reversal hopefully would eliminate any remaining liquid droplets.

The electric vaporizer dimensions were chosen to correspond to one of the steam vaporizers so that a direct comparison between vaporizers could be made. However, the impingement chamber was not used with the electric vaporizer. This vaporizer consisted of thin wall (0.012 in (0.30 mm)) stainless steel tubing with electrical leads attached at each end. Since the tubing was used as the resistive heater a high amperage (18 amps) low voltage (4.3 volts) direct current was used. To prevent possible shorts, the vaporizer tube was epoxied into micarta couplings at each end.

Initially, the accumulator volume was two liters but the first day's test results showed that the accumulator was too small. Thereafter, a four liter accumulator was used.

Analysis of the LNG used in the first flow facility test showed relatively high concentration of C_6^+ . To obtain a more representative LNG mixture, the LNG for this test was purchased from San Diego Gas and Electric Company. However, even this LNG contained approximately 0.08 mol percent C_6^+ .

For sample analysis, column configuration A was used. Prepared mixture H (table 4.2) was used as the calibration gas. Both integrators A and B were connected to gas chromatograph II so that their results could be compared.

5.2.3.1. Test results

A total of seven tests were made in the flow facility over a four day period. Table 5.18 lists the pertinent operating parameters and lists a comparison of measured and computed heating values and specific gravities. The computed quantities are real gas values computed from the compositions using the A.G.A. methods [1]. Table 5.19 gives the average values and estimated standard deviation of the compositions and the liquid densities computed using the extended corresponding states method [3].

Table 5.18. Operating variables and a comparison of measured and computed heating values and specific gravities for flow facility test 2.

Test	Vaporizer	Sampling Rate slpm	Number of Points	Heating Value, Btu/scf (MJ/m ³)		Specific Gravity		Remarks
				Measured	Computed ¹	Measured	Computed ¹	
A	1	14	3	1159.5 ⁶ (43.192)	1158.6 (43.159) 3.9 (0.145)	0.650	0.647 0.002 0.650 0.003	Two liter sample accumulator used in this test.
		19	6	1159.5 ⁵ (43.192)	1162.2 (43.293) 4.7 (0.175)	0.650		
	2	13	4	1159.5 ⁶ (43.192)	1163.7 (43.349) 7.9 (0.294)	0.650	0.651 0.005	
		16	3	1159.5 ⁵ (43.192)	1161.0 (43.248) 1.4 (0.052)	0.650	0.649 0.001	
B ²	2	13	7	1171.5 ⁶ (43.639)	1170.6 (43.606) 0.8 (0.030)	0.658	0.655 0.0007	Increased accumulator volume to 4L
		22	13	1169.0 ⁶ (43.546)	1169.7 (43.572) 0.6 (0.022)	0.658	0.654 0.0005	
C ³	1	16	5	1174.0 ^{4,6} (43.733)	1173.9 (43.729) 1.4 (0.052)	0.680	0.674 0.0007	Samples taken at 15 minute intervals after start-up of sampling system
D ³	1	6.0	3	1184.5-1186.0 ⁶ (44.124-44.180)	1178.6 (43.804) 6.9 (0.257)	0.680- 0.690	0.675 0.001	
		9.8	4	1176.5-1178.0 ⁶ (43.826-43.882)	1174.6 (43.755) 0.7 (0.026)	0.680	0.674 0.0	
	2	4.9	2	1184.5-1186.0 ⁶ (44.124-44.180)	1172.4 (43.673) 5.8 (0.216)	0.680- 0.690	0.674 0.004	
		9.5	4	1176.5-1178.0 ⁶ (43.826-43.882)	1175.8 (43.788) 1.3 (0.048)	0.680	0.674 0.0008	
E ²	1	19	4	1182.5 ⁵ (44.049)	1183.8 (44.098) 0.1 (0.004)	0.717	0.707 0.0	Had needle valve (valve S2, Fig. 5.21) wide open

Table 5.18. (continued)

Test	Vaporizer	Sampling Rate slpm	Number of Points	Heating Value, Btu/scf (MJ/m ³)		Specific Gravity		Remarks
				Measured	Computed ¹	Measured	Computed ¹	
F ²	1	16.8	3	1182.0-1183.0 ⁷ (44.031-44.068)	1184.2 (44.112)	0.717	0.707	Put electric vaporizer in system
	3	15.3	8	1182.0-1183.0 ⁷ (44.031-44.068)	0.8 (0.030) 1184.6 (44.127) 0.6 (0.022)	0.717	0.0006 0.707 0.0005	
G ²	1	16.9	4	1186.5 ⁷ (44.198)	1197.3 (44.600) 4.2 (0.156)	0.715	0.706 0.005	Removed insulation from sample line to vaporizer 1

¹Values given are the average values and the estimated standard deviations computed from the composition using the A.G.A. method [1].

²Integrator A results used in this test.

³Integrator B results used in this test.

⁴This is the steady state value; the value at the beginning of the test was 1188 Btu/scf (44.254 MJ/m³) and decreasing.

⁵Flow from vaporizer 1 to calorimeter and gravitometer.

⁶Flow from vaporizer 2 to calorimeter and gravitometer.

⁷Flow from vaporizer 3 to calorimeter and gravitometer.

Table 5.19. Average values and estimated standard deviations in composition and liquid density for the second flow facility test.

Test	Vaporizer	Sampling Rate slpm	Average Value and Estimated Standard Deviation in Composition, mol %									Liquid Density g/cm ³
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	C ₆ ⁺	
A ¹	1	14	0.0	84.997 0.413	12.005 0.334	2.414 0.067	0.190 0.005	0.226 0.006	0.048 0.0004	0.037 0.001	0.083 0.0008	0.4593 0.0015
		19	0.0009 0.002	84.609 0.486	12.324 0.390	2.469 0.077	0.193 0.006	0.232 0.007	0.049 0.002	0.038 0.002	0.084 0.003	0.4607 0.0016
	2	13	0.0	84.454 0.831	12.439 0.673	2.506 0.132	0.196 0.010	0.233 0.013	0.049 0.002	0.039 0.002	0.084	0.4610 0.0022
		16	0.0	84.669 0.080	12.302 0.019	2.466 0.080	0.187 0.004	0.220 0.005	0.044 0.0008	0.035 0.001	0.077 0.001	0.4603 0.0006
B ¹	2	13	0.007 0.005	83.772 0.075	12.968 0.064	2.604 0.013	0.205 0.001	0.244 0.001	0.052 0.0008	0.058 0.001	0.090 0.001	0.4537 0.0005
		22	0.011 0.003	83.866 0.050	12.904 0.037	2.585 0.016	0.203 0.002	0.242 0.002	0.052 0.0008	0.057 0.001	0.090 0.001	0.4534 0.0005
C ²	1	16	1.611 0.024	80.026 0.118	14.712 0.119	2.959 0.018	0.293 0.006	0.345 0.007	0.067 0.002	0.053 0.002	0.114 0.006	0.4802 0.0038
D ²	1	6.0	1.444 0.424	79.903 0.302	14.932 0.241	3.012 0.043	0.238 0.003	0.280 0.005	0.052 0.006	0.042 0.002	0.095 0.008	0.4797 0.0012
		9.8	1.611 0.024	80.026 0.118	14.712 0.119	2.959 0.018	0.233 0.002	0.274 0.003	0.051 0.001	0.042 0.002	0.090 0.005	0.4768 0.0010
	2	4.9	1.665 0.718	79.978 0.819	14.771 0.086	2.889 0.036	0.228 0.013	0.269 0.002	0.050 0.003	0.040 0.004	0.102 0.009	0.4795 0.0007
		9.5	1.491 0.024	80.103 0.132	14.757 0.082	2.956 0.025	0.234 0.014	0.275 0.003	0.052 0.003	0.042 0.002	0.089 0.005	0.4770 0.0008
E ¹	1	19	4.122 0.008	74.153 0.013	17.161 0.013	3.662 0.006	0.291 0.0003	0.347 0.003	0.077 0.0007	0.054 0.0006	0.133 0.001	0.5015 0.0010
F ¹	1	16.8	4.138 0.018	74.083 0.062	17.202 0.059	3.669 0.015	0.292 0.001	0.349 0.001	0.077 0.0002	0.055 0.0004	0.134 0.0003	0.5020 0.0010
	3	15.3	4.043 0.026	74.223 0.049	17.174 0.038	3.654 0.020	0.292 0.0006	0.348 0.001	0.077 0.0003	0.054 0.0005	0.134 0.0005	0.5020 0.0012
G ¹	1	16.9	3.231 0.279	74.597 0.129	17.517 0.139	3.729 0.019	0.298 0.003	0.356 0.002	0.078 0.001	0.056 0.0006	0.136 0.002	0.5002 0.0005

¹Integrator A results used in this test.

²Integrator B results used in this test.

Test A

Because the sampling rates would be comparable to those used in laboratory tests, two liter accumulators were used in test A. However, the test results suggested that the accumulator was too small. Figure 5.22 shows the deviation from the average heating value for this test. At the sample rates used in this test the accumulator residence time ranged between 10 and 18 seconds; this same range gave good results in laboratory tests. All remaining tests were made using four liter accumulators.

Test B

As shown in figure 5.23, results of test B clearly confirmed the need for the larger accumulator. Possible reasons for this include:

- a) the use of a constant wall temperature vaporizer (as opposed to the constant heat flux vaporizer used in the laboratory apparatus), or
- b) the presence of a finite heat leak into the sample line.

Unfortunately, we did not have sufficient time to identify the reason for needing the longer residence time.

Two points were eliminated from figure 5.23 and from the statistical analysis of test B results. For no obvious reason, one sample had a methane content five standard deviations higher than the average value for the test (both integrators gave the same result). However, the second outlier was the first sample taken after changing the sampling rate; it was taken less than 20 minutes after the change. This suggested the possibility of flow transients affecting the sampling precision.

Test C

Test C was a short test to determine if sampling system startup transients affected sampling precision. Figure 5.24 shows the deviation in heating value for this test. The deviations are relative to the average values obtained under steady conditions in test D which was run on the same day. In this test, samples were taken every 15 minutes after putting the sampling system in operation. Although the scatter was greater, especially in the nitrogen, methane and ethane contents, there was no obvious trend with time. Another sample was rejected in Test D which was one percent higher than the average heating value in that test; it too was drawn less than 30 minutes after changing sample rates. These results suggest that the sampling rate should be established at least 30 minutes before taking a sample.

Test D

Test D was made to determine the sampling precision of the two steam vaporizers at low sampling rates. The initial sampling rates were 9.8 and 9.5 slpm in vaporizers 1 and 2, respectively. At these rates neither the gravitometer nor the calorimeter indicated the precision being diminished for either vaporizer. These sampling rates corresponded to a linear liquid

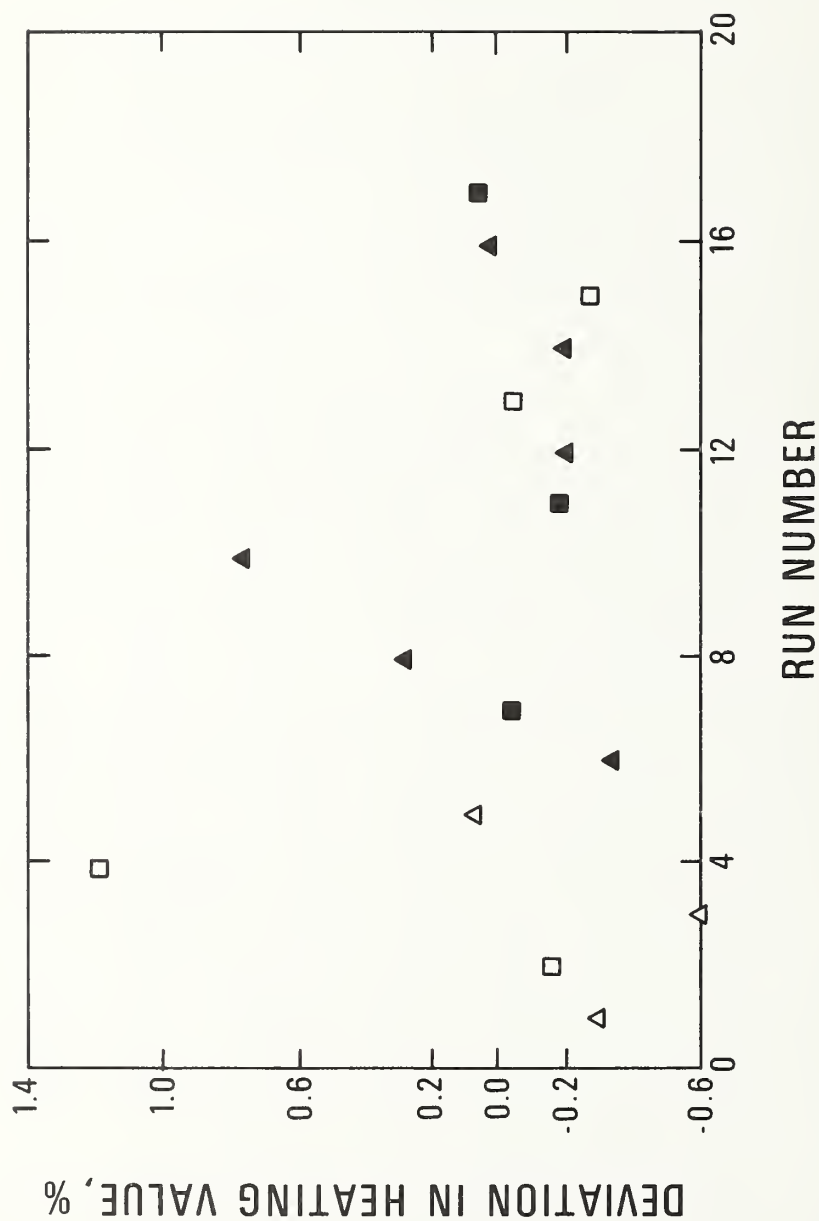


Figure 5.22. Deviations in the computed real gas heating value for flow facility test A. The symbols Δ and $\blacktriangle$ denote vaporizer 1 samples taken at 14 and 19 slpm while $\square$ and $\blacksquare$ denote vaporizer 2 samples taken at 13 and 16 slpm, respectively.

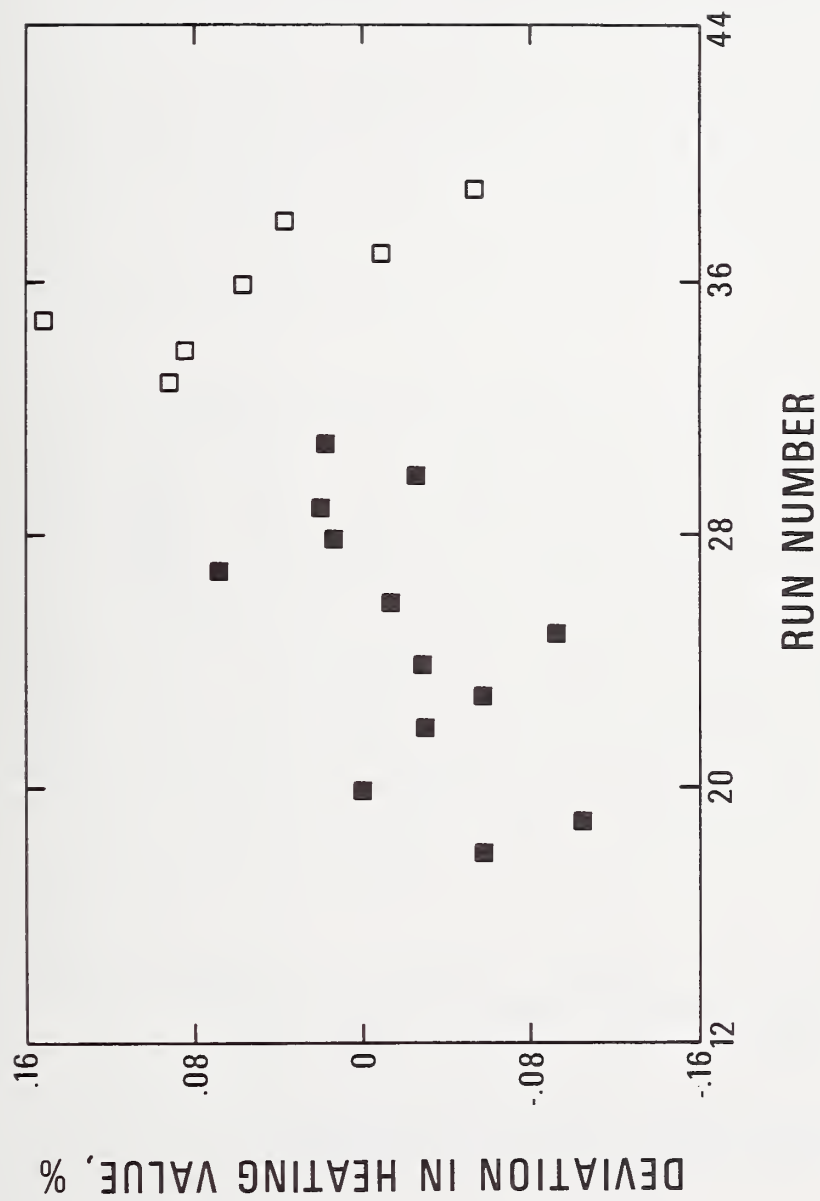


Figure 5.23. Deviations in computed real gas heating values for flow facility test B. The symbols $\square$ and $\blacksquare$ denote vaporizer 2 samples taken at 13 and 22 slpm, respectively.

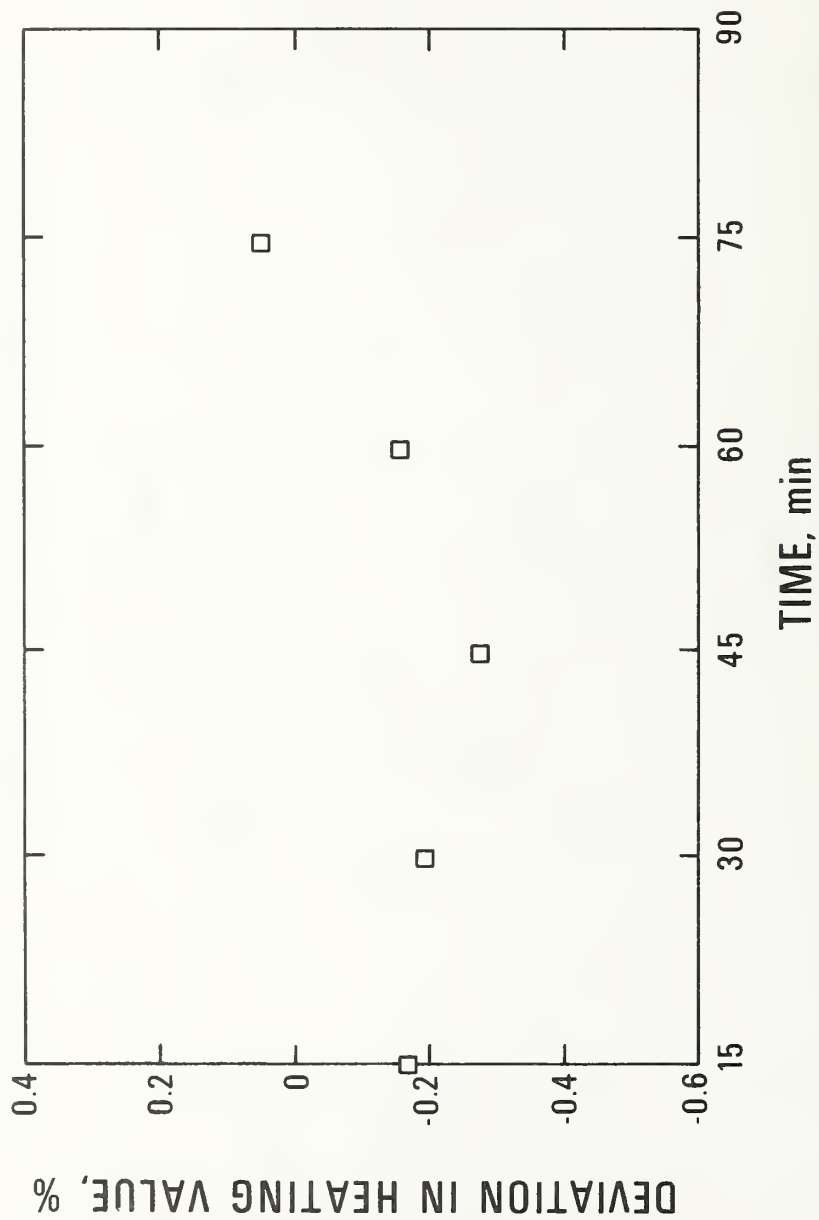


Figure 5.24. Deviations in the computed real gas heating values for flow facility test C.

flow rate of 4.0 cm/s and 1.6 cm/s in vaporizer 1 and 2, respectively. Next, the sampling rates were lowered so that the linear flow rates were 2.4 cm/s and 0.8 cm/s in vaporizers 1 and 2, respectively. For both liquid vaporizers these rates the gravitometer indicated a poorer sampling precision but the calorimeter did not. Figure 5.25 shows the change in precision of the computed heating value when going to the lower sampling rate. If the linear flow rate in the sample vaporizer was the important factor in determining sampling precision at low sample rates, the vaporizer 2 results at 9.5 slpm would have been much poorer than actually observed. This suggests that the poor precision at low sample rates originates at some point in the sampling system, other than in the vaporizer. It seemed plausible that the low sample flows could have been too low to provide adequate mixing in the accumulator. However, this probably was not the case because the accumulator residence time was 68 s in these tests; good results were obtained during shipboard tests with residence times of 120 s. However, the shipboard accumulator was heated to induce mixing.

Test E

Figure 5.26 shows the deviation in computed heating values for test E; this was a short test to see if the needle valve is necessary in a sampling system to prevent backflow. In this test the needle valve (valve S2, figure 5.21) was fully open. These results confirmed the laboratory results in that a needle valve is not necessary, at least when using a tube vaporizer where there is a finite pressure drop in the vaporizer. (However, the needle valve is useful for controlling sampling rates.) The pressure difference between the test section and sample panel varied between 0.5 psi (3 kPa) and 1.5 psi (10 kPa) during this test; when using the needle valve to control the sample rate the pressure difference was typically 20 psi (0.14 MPa) to 35 psi (0.24 MPa). (No significance should be put on the unusually high precision of this test; these estimated standard deviations are smaller than that of analysis alone.)

Test F

Test F was a comparison of electric (vaporizer 3) and steam (vaporizer 1) vaporizers. Figure 5.27 shows the deviation in computed heating value for this test. In this test the electric vaporizer had a heat input of 77 watts; at this power level, roughly half of the vaporizer was frostfree and the outlet temperature was roughly 100°F (38°C). As expected, the precision of the steam and electric vaporizers was comparable.

Test G

The last test considered the effect of heat leak on precision. Up to this point the sample lines had been insulated with fiberglass pipe insulation. During operation the lines felt slightly cool to the touch in most

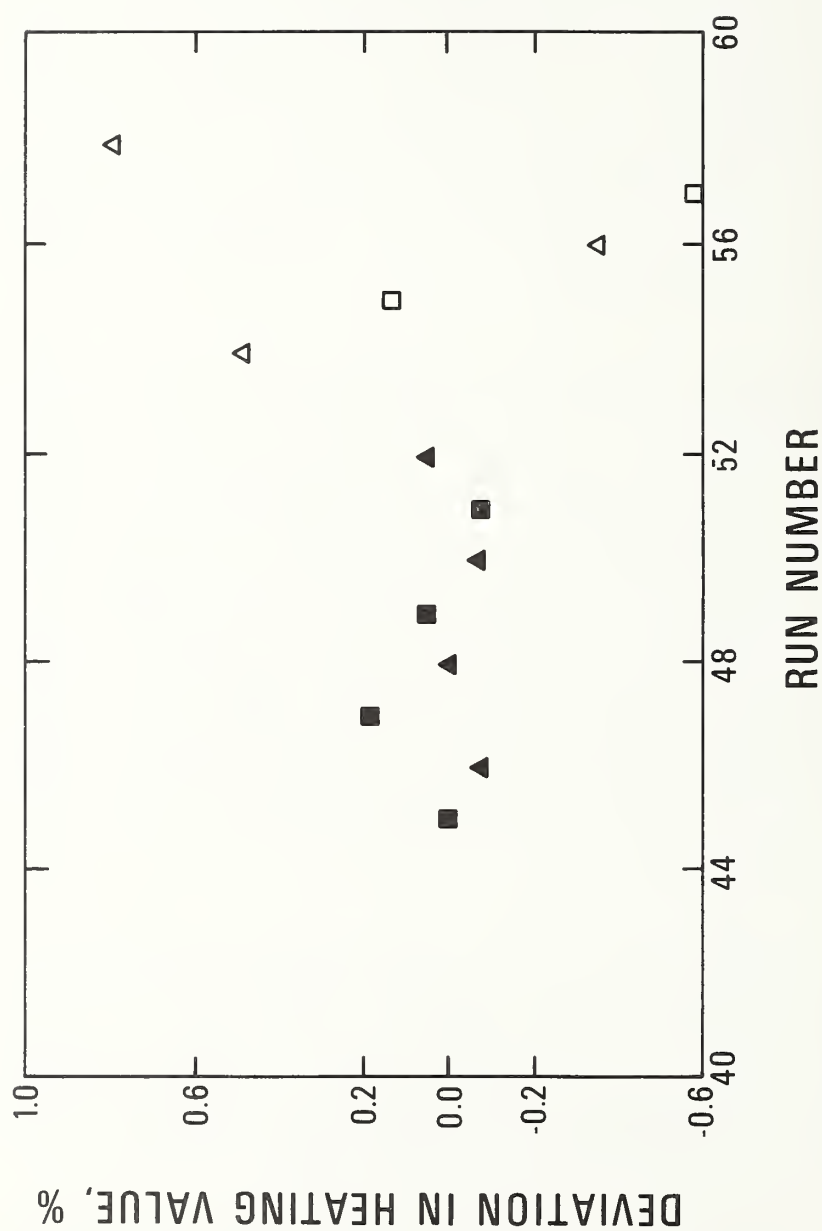


Figure 5.25. Deviations in the computed real gas heating values for flow facility test D. The symbols Δ and $\blacktriangle$ denote vaporizer 1 samples taken at 6.0 and 9.8 slpm while $\square$ and $\blacksquare$ denote vaporizer 2 samples taken at 4.9 and 9.5 slpm, respectively.

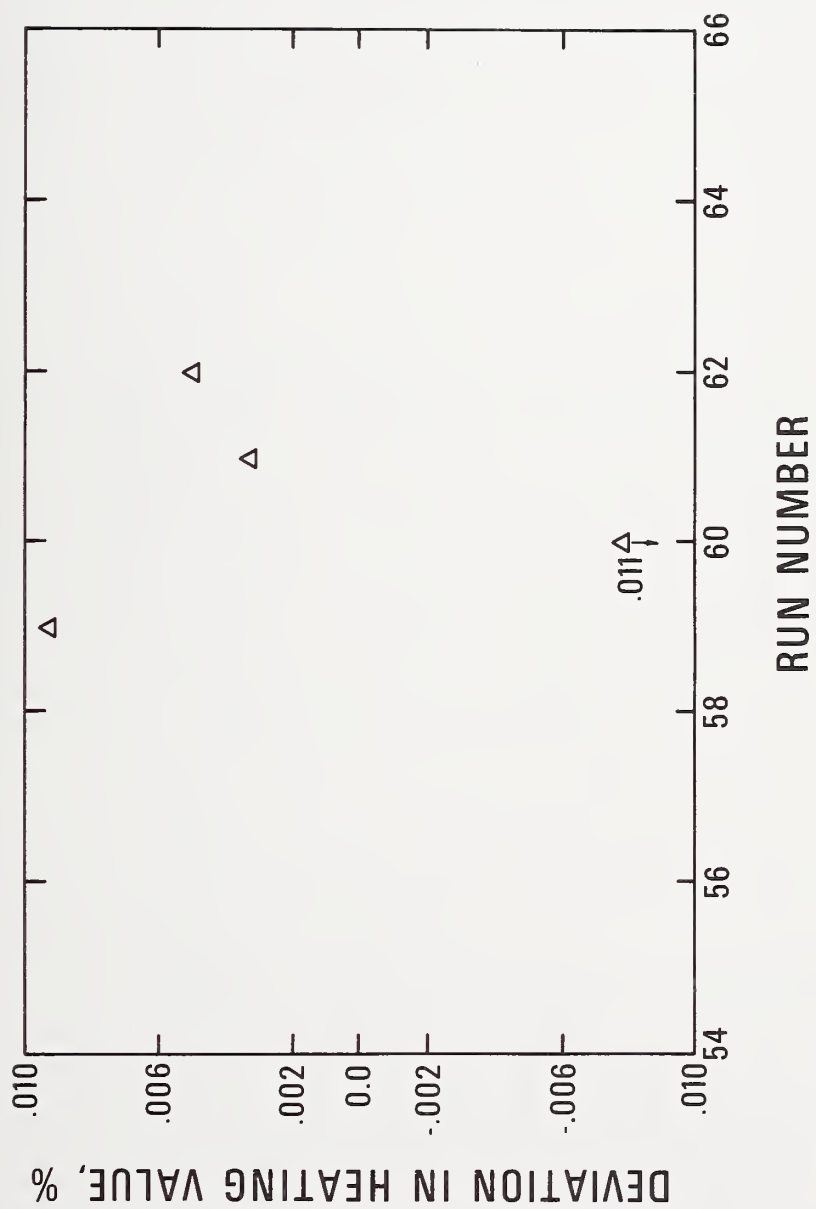


Figure 5.26. Deviations in the computed real gas heating values for flow facility test E.

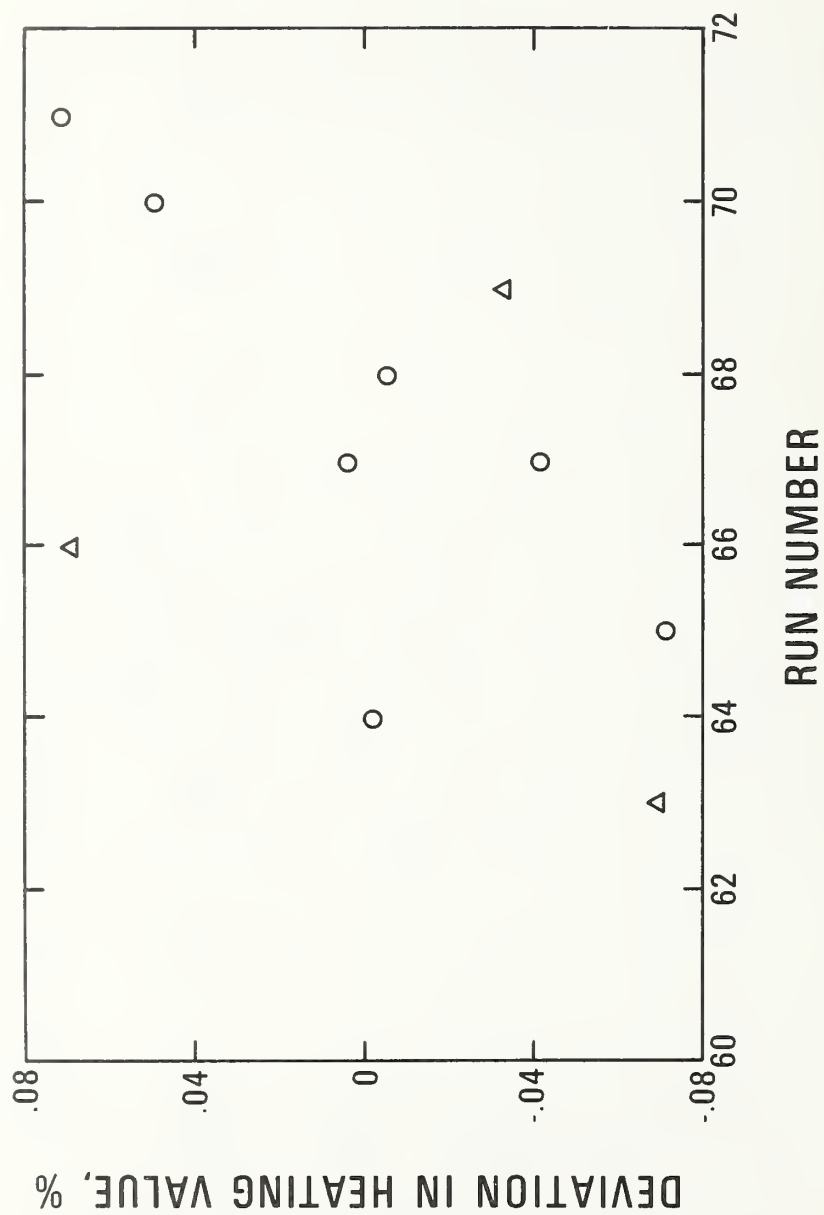


Figure 5.27. Deviations in the computed real gas heating values for flow Facility test F. The symbols Δ and O denote samples from vaporizers 1 and 3, respectively.

places; at the 90° bend between the block valve (Valve S1) and needle valve (Valve S2) there was a frost spot on the line to vaporizer 2 (or 3). No frost spots appeared on the sample line to vaporizer 1. For this test, the insulation was removed from the sample line to vaporizer 1. Samples from this vaporizer were going to the calorimeter and gravitometer during the insulation removal. Before all of the insulation had been stripped away the calorimeter went off scale, indicating a heating value of greater than 1195 Btu/scf (44.515 MJ/m³). As figure 5.28 shows, the computed heating values were significantly higher for the uninsulated sampling system. (The deviations are referenced to the average values obtained in test F.) The sample analyses indicated a 0.8 mole percent reduction in nitrogen which was statistically significant. Also, there was a 0.3 mole percent increase in the ethane content; but based on the limited number of samples, this increase could not be considered statistically significant. This indicates that backflashing occurred in the sample line and that it enriched the heavy components in the observed samples.

General Comments

Based on the shipboard tests, we expected frost to accumulate at the 90° bend on the steam vaporizer sample outlet lines at sample rates above 10 slpm. The impingement chamber apparently worked satisfactorily because there never was any indication of frost at the bend. The sample lines were felt at and near the bends several times throughout the tests; there was never an indication that the line was cooler at the bend.

Throughout these tests, the comparison between the measured and computed heating values was fairly good considering that the C₆+ fraction ranged between 0.08 and 0.13 mol percent. An analysis of the heavy fraction indicated that it had roughly equal parts of C₆'s and C₇'s. Therefore, the response factor for C₆+ peaks, K_{C₆+}, was computed by

$$K_{C_6+} = K_{nC_4} (D_{nC_6} + D_{nC_7}) / 2D_{nC_4} \quad (5.1)$$

where K_{nC₄} is the measured response factor for normal butane and the D's are the response factor reported by Dietz [25].

Except for runs E, F and G (the final day's tests), the agreement between measured and computed specific gravities was excellent. It is not known why the final day's results were consistently low by 1.3 to 1.4 percent, especially since the heating values agreed to 0.14 percent or better.

The estimated standard deviation in the computed liquid densities listed in table 5.19 is much larger than observed in previous tests. However, the densities in the flow facility tests reflected both temperature and composition fluctuations. The temperature fluctuations, which were as large as 1.5 K, make comparisons based on density meaningless.

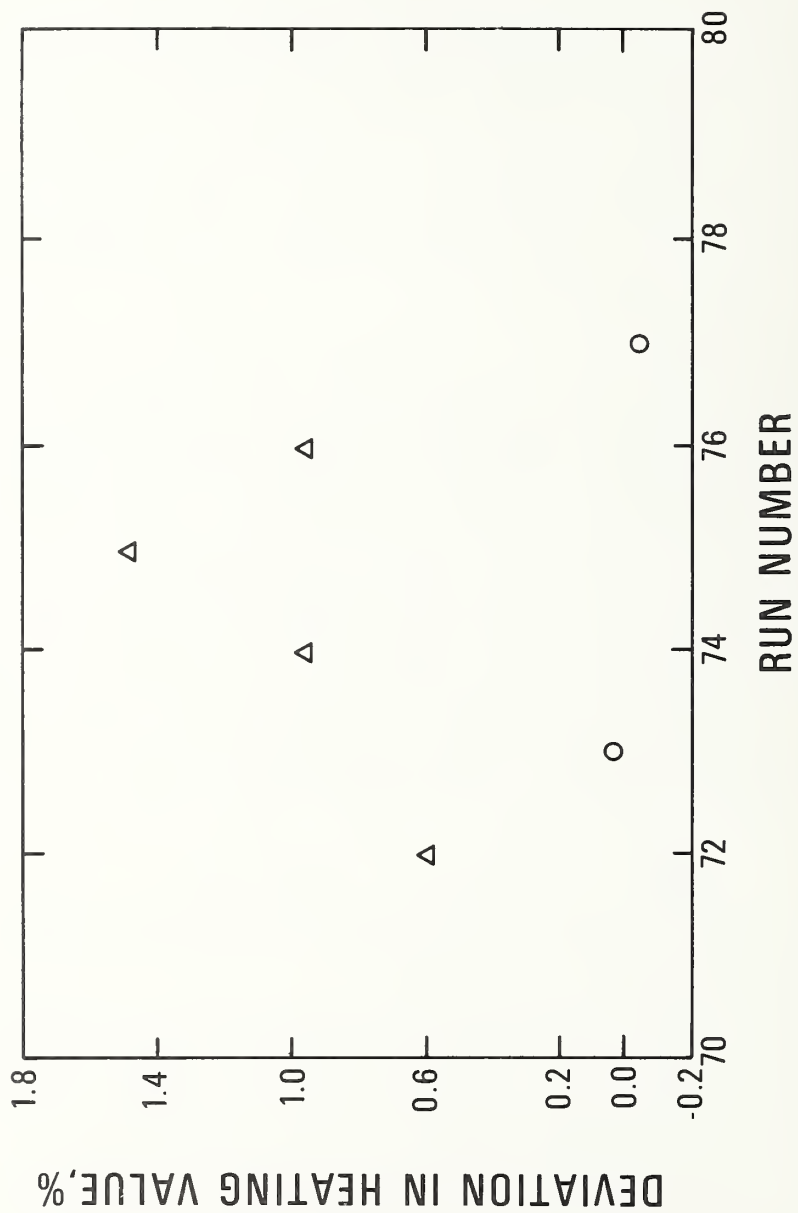


Figure 5.28. Deviations in the computed real gas heating values for flow facility test G. The symbols Δ and O denote samples from vaporizers 1 and 3, respectively.

Throughout these tests, integrator A had difficulties in reliably measuring the C_6+ peak area because the peak had a small peak height and wide peak width compared to the other peaks (see section 4.3.1.1 for more details). Except for the first day integrator B reported consistent C_6+ peak areas. Therefore, the C_6+ peak area for integrator A was computed by assuming that the ratio of the C_6+ peak area to that of the normal butane peak area was equal for both integrators; this ratio for integrator B was constant to within ± 2 percent throughout the tests. (A check showed that the ratios of iso to normal butane peak areas for the two integrators were equal to within one percent or better.)

During the first two days the two integrators reported comparable compositions for all components except iso and normal pentane; this difference was traced to calibration problems with integrator B. The typical spread between the calculated heating values for the two integrators was 0.05 to 0.14 percent; the difference in pentane contents reported by the two integrators accounted for this heating value difference.

On the third day (Test D) the program in integrator A was altered to hopefully eliminate the scatter in detecting the C_6+ peak. This change not only did not improve the C_6+ peak detection but it caused increased scatter in integrating the nitrogen and methane peaks. (In this chromatographic column arrangement, nitrogen and methane eluted after C_6+ .) The differences in computed heating values differed by as much as one percent with integrator A showing the most scatter.

On the last day the integrator A program was identical to the program used the second day. On this day integrator B gave erratic nitrogen contents even though no program changes were made. However, we noted that the peak width parameter (which was automatically updated after each peak) varied between 20 and 66 seconds. This variation affected the next (nitrogen) peak area measurement but not the methane peak area measurement. This is because the typical peak widths for nitrogen and methane were 20 and 60 s, respectively. Agreement between the two integrators occurred only when the updated peak width parameter was between 20 and 30 s.

5.3. Conclusions and Recommendations

This work has systematically considered many factors which could affect the precision and accuracy of the composition of samples withdrawn from a flowing LNG stream. Laboratory and field tests showed that it is feasible to obtain and analyze representative samples with a precision (based on three standard deviations) of better than ± 0.30 percent in the computed heating value; this includes the gas analysis precision of ± 0.06 percent. The accuracy of the measurement depends only on the gas analysis accuracy; both laboratory and shipboard tests showed that a properly designed and operated sampling system produces no statistically significant bias. A

comparison of measured and computed heating values during the second flow facility test verified that the difference between the two values was within the combined uncertainty of the calorimeter measurement (± 0.3 percent [30]) and the sampling measurement process.

Table 5.20 summarizes the variables evaluated in the sampling tests and groups them according to their effect on sampling precision and accuracy. The test results indicated that heat leak causes backflashing of nitrogen (and probably methane) which enriches the samples in the heavy (C_2+) components. However, field tests showed that insulating the sample line with one inch (25 mm) thick layer of fiberglass pipe insulation sealed with duct tape eliminates the heat leak problem.

Table 5.20. Variables evaluated for their effect on LNG sampling precision and accuracy.

Variables affecting both sampling precision and accuracy:

- Heat leak to liquid sample line

Variables affecting sampling precision but not accuracy:

- Sampling rate
- Vaporizer design
- Time-averaging the vaporized sample
- Sampling rate transients
- Probe design

Variables not affecting sampling precision and accuracy:

- Temperature and pressure at the sampling point
- Pressure differential between the sampling point pressure and the equilibrium LNG pressure
- Flow rate past the sampling point
- Pressure drop in the liquid sample line between the sampling point and vaporizer
- Composition of the liquid being sampled.

Five variables were found to adversely affect sampling precision. Both laboratory and field tests showed that below a certain sampling rate, the sampling precision diminishes; this minimum sampling rate varied among various sampling system designs. A mechanism for the sampling rate's importance is unknown. Attempts to correlate the sampling rate with heat leak, accumulator residence time, linear flow rate in the liquid sample line and linear flow rate in the vaporizer failed. However, criteria for setting the minimum allowable sample rate must be found before general guidelines for LNG sampling system design can be established. The next section gives sampling rates for the recommended sampling system design.

Laboratory results clearly indicated that a small diameter tube vaporizer was better than a vaporizer with a large cross sectional area. Tests also showed that rapid sample vaporization gave the best results. However, there is no advantage in having the vaporizer outlet temperature above 100°F (38°C). Both steam and electrically heated vaporizers produce comparable results.

The need to time-average the sample stream leaving the vaporizer in an accumulator arises from fractionation during vaporization. The minimum residence time necessary to obtain precise results depended upon the sampling system; residence times of 20 to 120 s gave good results.

Although the field tests were inconclusive, they indicated that taking samples within 30 minutes of a major change in sampling rate diminishes sampling precision. However, minor fluctuations and slow drifts in sampling rates did not affect the results.

The side tap probe was found to be more reliable than an upstream facing pitot tube. For unknown reasons the pitot tube occasionally gave erratic results.

The conclusion that certain variables did not affect sampling error must be considered valid only over the ranges tested; however, this range usually included the anticipated operating ranges of on and off-loading of LNG tankers.

There is no reason to expect the temperature and pressure to affect sampling error provided the conditions are far from the critical point of the LNG.

At some low value the difference between the pressure of the sampled liquid and the saturation pressure of the LNG (i.e., subcooling) in combination with heat leak should create premature vaporization which could adversely affect sampling precision; however, this effect was not detected at pressure differentials as low as 3.9 psi (25 kPa) (this corresponds to ~ 0.5 K subcooling).

LNG flow rates corresponding to Reynold's numbers ranging between 1500 and 50,000 did not affect sampling precision; it is unlikely that there would be any adverse effect on precision at the higher Reynold's numbers anticipated in on and off loading operations.

The fact that a pressure drop in the liquid sample line which is comparable to the pressure drop in the vaporizer is unnecessary eliminates the need for a liquid flow control valve. However, this conclusion is known to be valid only when using a tube vaporizer.

Finally, there is no problem in sampling LNG mixtures which contain up to 0.15 percent C_5+ . The heavies would be a problem if they raised the sample dew point temperature to near ambient temperature.

5.3.1. Recommended LNG Sampling System Design and Operating Criteria

Based on the laboratory and field test results, the following sampling system design criteria are recommended:

Probe - side tap mounted horizontally with the probe flush with the wall of the LNG pipe; probe inner diameter should be as small as feasible (< 0.25 inch (6.4 mm)).

Liquid phase needle valve - optional, but useful for flow control.

Liquid sample line - use as small a bore tubing as feasible (≤ 0.20 inch (5.1 mm)) and make the line as short as possible. (A filter is recommended to prevent line plugging.)

Sample vaporizer - use a coiled tube vaporizer with the tube inner diameter comparable to the sample line tubing. If steam is the vaporizing medium, use the design procedure outlined in section 5.2.2 with an impingement chamber. Electrically heated vaporizers can be designed using the same procedure as steam vaporizers; however, thin walled tubing is necessary to minimize the necessary amperage. Also, a thermostatic control is necessary to prevent the vaporizer from overheating if the sample flow rate decreases.

Accumulator - size for a residence time of at least 45 s; the length to diameter ratio of the accumulator should be greater than 1.5. The sample inlet line should extend to near the bottom of the vessel but the outlet line should not penetrate the vessel volume. Heating the bottom of the accumulator may produce improved mixing.

Insulation - the probe and liquid sample line should be insulated sufficiently to prevent moisture from condensing on the lines.

The following sample system operating conditions are within the range of values found to give good results in the field tests and therefore are recommended:

Sampling rate - greater than 20 slpm; this criteria applies only to the recommended sampling system.

Sample pressure - preferably greater than 20 psig (0.24 MPa) to provide sufficient gas sample for purging and replicate analyses from a 500 cm³ sample cylinder.

Vaporizer outlet temperature - 80 to 100°F (27 to 38°C).

The shipboard tests and flow facility test 2 sampling systems both worked satisfactorily and may be considered proven sampling systems.

5.3.2. Recommendations for Future Work

All tests clearly indicated that sampling rate is an important operating variable but no criteria for establishing the minimum acceptable rate were found. The existing test results need to be carefully reexamined for clues to understanding how sampling rate affects sampling precision. Hopefully, this would provide the answer, or would at least suggest future tests.

At this point, the need for time averaging denotes the lack of understanding of the sample vaporization process. Different vaporizer designs

should be tested with the goal of understanding the vaporization process and eliminating the need for an accumulator, if possible. (These tests probably would provide some insight into the sample rate problem too.)

Resolving the uncertainties associated with the sampling rate and vaporizer design will likely allow establishment of general design and operating criteria. At this point a standard procedure should be developed for continuously sampling LNG from flowing streams.

Other types of LNG sampling techniques need to be studied. There should be additional work on systems for batch sampling LNG, e.g., Cook sampler. Also, reliable systems for sampling LNG from tanks need to be developed.

The goal of all future work should be to establish the basis for standard LNG sampling procedures which have an uncertainty comparable to that obtained from gas analysis alone.

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APPENDIX 7.1. GAS ANALYSIS DATA

This appendix lists the compositions and computed ideal gas heating values obtained during the evaluation of gas analysis techniques. The heating values were computed using the values listed in table 2.2. To convert from MJ/m³ to BTU/SCF multiply by 26.845. Unless stated otherwise, all peak areas were measured using integrator A. Mix in these tables refers to the prepared mixtures listed in table 4.2.

7.1.1. Comparison Between Mass Spectrometry and Gas Chromatography.

Table 7.1.1 lists the mass spectrometry analyses obtained from a local laboratory. The table lists the reported duplicate analyses on each sample. Table 7.1.2 lists the gas chromatograph analyses obtained using the two column configuration.

Table 7.1.1. Results of mass spectrometer analyses.

Sample ID	Mix	Composition, mol %						Heating Value MJ/m ³
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	
441	B	5.62	84.42	8.34	.35	.70	.56	39.207
441	B	3.42	86.61	8.61	0.00	.80	.55	39.394
445	B	1.81	87.55	8.49	.98	.62	.55	40.366
445	B	5.23	84.89	8.38	.25	.68	.56	39.295
442	A	8.53	76.72	4.21	5.97	2.59	1.99	42.980
442	A	6.91	78.49	4.73	4.92	2.93	2.01	43.358
444	A	8.45	76.69	4.22	6.24	2.45	1.95	42.918
444	A	7.58	77.57	4.25	6.14	2.47	1.99	43.250
443	D	1.16	84.03	4.93	5.74	.18	3.96	45.380
443	D	1.49	83.12	4.71	6.59	.04	4.06	45.528
446	D	7.09	78.22	4.43	6.19	.09	3.98	43.189
446	D	5.23	79.86	4.37	6.64	.01	3.88	43.985
501	B	2.35	87.77	8.48	0.00	.84	.56	40.391
501	B	1.50	88.20	8.95	0.00	.82	.53	40.312
503	B	1.83	88.13	8.59	.14	.74	.56	40.523
503	B	1.68	88.12	8.82	0.00	.82	.56	40.732
502	D	1.32	82.53	4.12	7.73	.07	4.23	46.339
502	D	1.23	83.01	4.46	7.00	.08	4.23	46.059
504	D	1.69	82.76	4.63	6.62	.03	4.28	45.726
504	D	1.23	83.32	4.52	6.62	.02	4.30	45.971
505	C	3.33	88.24	6.30	1.35	.57	.21	39.549
505	C	1.03	90.55	6.53	1.04	.65	.20	40.466
506	C	4.22	87.15	6.07	1.84	.50	.22	39.473
506	C	1.21	90.53	6.67	.67	.71	.21	40.285

Table 7.1.2. Results of gas chromatographic analyses.

Date	Mix	Composition, mol %						Heating Value MJ/m ³
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	
9/ 8	B	1.352	88.111	8.408	.958	.485	.686	41.098
9/ 8	B	1.349	88.141	8.401	.954	.477	.678	41.081
9/ 8	A	4.330	81.301	4.765	4.803	2.401	2.400	44.153
9/10	B	1.423	88.043	8.389	.980	.475	.690	41.073
9/10	B	1.260	88.204	8.406	.975	.474	.680	41.127
9/13	B	1.305	88.106	8.422	.991	.485	.691	41.142
9/13	B	1.296	88.116	8.417	.991	.485	.696	41.148
9/13	C	.666	90.514	6.041	2.170	.307	.302	40.896
9/13	C	.552	90.654	6.022	2.157	.306	.309	40.931
9/13	B	1.331	88.109	8.394	.985	.490	.691	41.125
9/13	B	1.336	88.094	8.399	.999	.485	.687	41.125
9/14	B	1.291	88.199	8.379	.978	.476	.677	41.109
9/14	B	1.297	88.189	8.379	.972	.476	.687	41.112
9/14	B	1.341	88.163	8.359	.976	.483	.678	41.090
9/14	A	4.503	80.947	4.736	4.921	2.432	2.462	44.224
9/14	A	4.159	81.465	4.745	4.872	2.385	2.375	44.216
9/14	A	4.056	81.507	4.756	4.899	2.393	2.390	44.293
9/14	A	4.042	81.600	4.727	4.872	2.381	2.378	44.255
9/14	A	4.031	81.564	4.734	4.889	2.391	2.390	44.289
9/14	B	1.255	88.241	8.383	.971	.475	.675	41.117
9/29	B	1.357	88.182	8.344	.971	.477	.668	41.064
9/29	B	1.347	88.103	8.403	.978	.491	.678	41.108
9/29	D	.009	85.342	5.086	4.873	0.000	4.690	45.828
9/29	D	.008	85.449	5.064	4.855	0.000	4.624	45.756
9/29	D	.008	85.386	5.065	4.872	0.000	4.670	45.804
9/29	D	.009	85.369	5.075	4.861	0.000	4.686	45.814
9/29	C	.644	90.505	6.033	2.171	.304	.323	40.929
9/29	C	.618	90.580	6.048	2.154	.295	.305	40.899
9/29	C	.681	90.562	6.020	2.143	.297	.297	40.857
10/ 1	E	1.382	85.887	8.504	2.990	.527	.710	42.313
10/ 1	C	.625	90.526	6.061	2.163	.310	.315	40.926
10/ 1	C	.626	90.562	6.044	2.155	.305	.308	40.907
10/ 1	C	.623	90.561	6.046	2.154	.306	.310	40.910
10/ 1	C	.620	90.538	6.066	2.164	.306	.306	40.919
10/ 1	D	.011	85.277	5.108	4.878	0.000	4.727	45.867
10/ 1	D	.008	85.166	5.104	4.897	0.000	4.826	45.961
10/ 1	D	.009	85.285	5.082	4.877	0.000	4.748	45.877
10/ 1	D	.008	85.291	5.086	4.877	0.000	4.738	45.871
10/ 1	E	1.375	85.877	8.495	3.010	.519	.724	42.329
10/ 4	E	1.391	85.993	8.432	2.956	.519	.709	42.263

Table 7.1.2. (Continued)

Date	Mix	Composition, mol %						Heating
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Value MJ/m ³
10/ 4	C	.645	90.525	6.043	2.168	.305	.315	40.912
10/ 4	C	.631	90.563	6.042	2.159	.299	.306	40.900
10/ 4	C	.632	90.571	6.034	2.153	.301	.309	40.898
10/ 4	C	.639	90.541	6.056	2.158	.301	.305	40.901

7.1.2. Statistical evaluation of gas chromatographic analyses.

Table 7.1.3 lists the data taken to determine the accuracy and precision of gas chromatographic analyses using the two column configuration. The first digit of the analysis number refers to the day while the remaining digits refer to the sequence number. In this table, mixtures A, B, C and D correspond to mixtures E, F, G and C in table 4.2. Table 7.1.3.A lists the outlying analyses which were not included in the statistical evaluation. Table 7.1.4 gives the results of repetitive analyses of mixture E which were made after the integrator was repaired (see section 4.2.).

Table 7.1.3. Raw data for determining the precision and accuracy of gas chromatographic analyses.

Analysis Number	Operator	Mix	Composition, mol %						Heating Value MJ/m ³
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	
101	2	D	.615	90.585	6.031	2.152	.308	.309	40.909
102	1	C	10.969	65.875	16.249	5.436	.496	.976	42.468
103	2	B	.974	88.224	7.257	2.560	.491	.494	41.660
104	1	A	1.383	85.921	8.480	2.978	.523	.715	42.300
105	2	C	10.980	65.878	16.240	5.439	.491	.973	42.457
106	2	A	1.384	85.910	8.487	2.980	.524	.715	42.304
107	1	B	.971	88.196	7.270	2.570	.495	.499	41.677
108	1	D	.614	90.585	6.040	2.150	.303	.309	40.906
109	2	D	.614	90.562	6.054	2.156	.305	.310	40.916
110	1	C	10.966	65.802	16.306	5.463	.500	.963	42.493
111	1	A	1.383	85.905	8.495	2.980	.526	.711	42.304
112	2	B	.970	88.219	7.260	2.572	.491	.488	41.664
113	2	A	1.385	85.893	8.484	2.988	.526	.724	42.316
114	1	B	.971	88.216	7.262	2.568	.493	.491	41.666
115	1	D	.613	90.589	6.037	2.154	.301	.305	40.904
116	2	C	10.980	65.839	16.280	5.444	.493	.965	42.466
117	1	A	1.380	85.931	8.468	2.995	.519	.707	42.297
118	1	C	10.969	65.876	16.249	5.439	.495	.972	42.466
119	2	D	.614	90.581	6.039	2.153	.299	.314	40.909
120	2	B	.971	88.194	7.277	2.568	.497	.493	41.675
121	2	C	10.964	65.791	16.313	5.466	.495	.970	42.500
122	2	A	1.384	85.921	8.491	2.978	.514	.712	42.292
123	1	D	.614	90.589	6.019	2.164	.303	.311	40.910
124	1	B	.971	88.206	7.271	2.564	.495	.493	41.669
201	1	A	1.381	85.919	8.494	2.976	.522	.708	42.297
202	2	B	.971	88.193	7.270	2.582	.496	.488	41.676
203	1	C	10.960	65.883	16.261	5.432	.499	.965	42.466
204	2	D	.614	90.561	6.043	2.157	.312	.313	40.922
205	2	A	1.382	85.918	8.488	2.981	.524	.707	42.298
206	2	C	10.970	65.842	16.280	5.449	.497	.961	42.473
207	1	D	.615	90.572	6.050	2.156	.304	.303	40.908
208	1	B	.969	88.197	7.272	2.562	.498	.502	41.679
209	1	A	1.382	85.915	8.497	2.977	.523	.706	42.297
210	2	D	.614	90.547	6.066	2.166	.302	.305	40.919
211	1	C	10.969	65.832	16.288	5.442	.498	.971	42.481
212	2	B	.971	88.213	7.270	2.568	.492	.486	41.663
213	2	C	10.971	65.855	16.281	5.444	.492	.956	42.462
214	2	A	1.384	85.909	8.481	2.993	.522	.711	42.304
215	1	D	.614	90.594	6.036	2.149	.302	.306	40.901
216	1	B	.969	88.203	7.273	2.564	.497	.494	41.673

Table 7.1.3. (Continued)

Analysis Number	Operator	Mix	Composition, mol %						Heating Value MJ/m ³
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	
217	2	D	.614	90.566	6.058	2.150	.307	.305	40.911
218	1	A	1.382	85.926	8.480	2.981	.523	.709	42.297
219	1	C	10.969	65.817	16.309	5.444	.496	.964	42.480
220	2	B	.971	88.199	7.270	2.573	.491	.496	41.673
221	2	C	10.970	65.841	16.270	5.459	.497	.963	42.477
222	1	B	.972	88.200	7.262	2.578	.494	.494	41.674
223	2	A	1.382	85.919	8.494	2.975	.521	.709	42.296
224	1	D	.613	90.573	6.037	2.165	.303	.310	40.915
301	2	D	.614	90.593	6.032	2.155	.304	.303	40.903
302	2	B	.972	88.195	7.279	2.562	.497	.496	41.674
303	1	C	10.956	65.740	16.360	5.480	.497	.967	42.523
304	1	A	1.385	85.919	8.494	2.980	.517	.705	42.291
305	2	C	10.989	65.815	16.289	5.453	.490	.965	42.467
306	1	B	.971	88.168	7.304	2.569	.497	.491	41.681
307	1	D	.615	90.565	6.049	2.154	.307	.311	40.916
308	2	A	1.383	85.888	8.506	2.983	.525	.715	42.312
309	2	B	.972	88.183	7.290	2.573	.492	.490	41.674
310	1	C	10.962	65.775	16.339	5.453	.504	.967	42.505
311	2	D	.613	90.557	6.052	2.165	.304	.309	40.919
312	1	A	1.381	85.893	8.504	2.984	.521	.717	42.311
313	2	A	1.383	85.870	8.527	2.988	.523	.709	42.314
314	1	B	.971	88.170	7.298	2.570	.497	.494	41.683
315	1	D	.614	90.555	6.053	2.170	.303	.306	40.919
316	2	C	10.980	65.823	16.281	5.452	.498	.966	42.476
317	2	D	.614	90.565	6.053	2.156	.300	.312	40.913
318	2	B	.969	88.175	7.297	2.570	.497	.493	41.682
319	1	A	1.384	85.877	8.509	2.984	.525	.721	42.318
320	1	C	10.970	65.850	16.280	5.438	.502	.960	42.470
321	1	D	.617	90.565	6.052	2.152	.306	.308	40.911
322	2	A	1.383	85.931	8.479	2.982	.521	.704	42.291
323	2	C	10.965	65.837	16.274	5.461	.497	.966	42.483
324	1	B	.971	88.172	7.294	2.577	.498	.488	41.681
401	2	B	.968	88.217	7.264	2.564	.490	.497	41.668
402	1	C	10.980	65.849	16.250	5.459	.500	.963	42.470
403	1	A	1.383	85.915	8.484	2.986	.523	.709	42.301
404	2	D	.616	90.589	6.024	2.166	.303	.302	40.904

Table 7.1.3. (Continued)

Analysis Number	Operator	Mix	Composition, mol %						Heating Value MJ/m ³
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	
405	1	B	.971	88.206	7.272	2.568	.495	.489	41.668
406	2	A	1.383	85.929	8.464	2.978	.532	.714	42.302
407	2	C	10.979	65.863	16.248	5.433	.496	.980	42.466
408	1	D	.614	90.571	6.039	2.155	.306	.315	40.917
409	2	B	.970	88.186	7.264	2.568	.505	.507	41.690
410	1	C	10.965	65.794	16.264	5.496	.505	.976	42.516
411	1	A	1.383	85.894	8.487	2.982	.530	.724	42.318
412	2	D	.614	90.562	6.042	2.167	.303	.312	40.919
413	2	A	1.383	85.924	8.471	2.988	.524	.710	42.300
414	1	D	.613	90.568	6.044	2.155	.306	.314	40.917
415	1	B	.971	88.204	7.265	2.571	.496	.492	41.672
416	2	C	10.976	65.868	16.245	5.447	.495	.969	42.464
417	2	B	.971	88.185	7.270	2.591	.495	.488	41.680
418	2	D	.615	90.583	6.036	2.153	.306	.308	40.908
419	1	C	10.979	65.846	16.249	5.453	.504	.969	42.475
420	1	A	1.383	85.905	8.498	2.984	.523	.707	42.302
421	1	B	.972	88.200	7.268	2.568	.495	.497	41.674
422	2	A	1.382	85.927	8.474	2.982	.526	.710	42.299
423	2	C	10.981	65.866	16.231	5.457	.503	.961	42.464
424	1	D	.615	90.577	6.042	2.156	.302	.308	40.908

Table 7.1.3.A. Outlier analyses not used in statistical evaluation.

Analysis Number	Operator	Mix	Composition, mol %						Heating Value MJ/m ³
			N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	
104	1	A	1.393	85.912	8.479	2.978	.523	.715	42.296
109	2	D	.328	90.846	6.055	2.156	.305	.310	41.024
110	1	C	10.620	66.129	16.320	5.468	.500	.964	42.631
121	2	C	10.690	66.041	16.330	5.472	.496	.971	42.613
202	2	B	.982	88.183	7.269	2.582	.496	.488	41.671
210	2	D	.444	90.715	6.068	2.167	.302	.305	40.984
217	2	C	.502	90.677	6.059	2.150	.307	.305	40.954
219	1	C	10.760	66.011	16.320	5.448	.496	.965	42.565
220	2	B	.918	88.253	7.269	2.573	.491	.496	41.693
303	1	C	10.400	66.252	16.390	5.490	.498	.969	42.749
310	1	C	10.680	66.028	16.360	5.460	.505	.968	42.623
317	2	D	.628	90.550	6.054	2.156	.300	.312	40.909
323	2	C	10.880	65.900	16.290	5.466	.497	.967	42.524
403	1	A	1.392	85.907	8.483	2.986	.523	.709	42.297
410	1	C	10.060	66.527	16.429	5.501	.506	.977	42.908
411	1	A	.594	86.591	8.547	3.014	.530	.724	42.650
415	1	B	.983	88.194	7.264	2.571	.496	.492	41.667
416	2	C	10.999	66.006	16.069	5.459	.496	.971	42.414
417	2	B	.945	88.208	7.272	2.592	.495	.488	41.691
420	1	A	1.390	85.899	8.497	2.984	.523	.707	42.299

Table 7.1.4. Raw data for determining the accuracy and precision of gas chromatographic analysis taken after integrator repair.

Analysis Number	Time	Composition, mol %						Heating Value MJ/m ³
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	
101	1117	1.383	85.925	8.477	2.982	.526	.707	42.297
102	1133	1.382	85.933	8.479	2.980	.521	.705	42.291
103	1149	1.382	85.904	8.487	2.989	.527	.711	42.308
104	1204	1.383	85.913	8.476	2.998	.523	.707	42.303
105	1219	1.381	85.903	8.504	2.976	.522	.714	42.305
106	1234	1.384	85.907	8.501	2.977	.521	.710	42.299
107	1250	1.383	85.908	8.492	2.979	.522	.716	42.304
108	1305	1.383	85.939	8.469	2.980	.521	.708	42.290
109	1321	1.378	85.960	8.458	2.971	.521	.712	42.287
110	1336	1.381	85.941	8.470	2.976	.521	.711	42.292
111	1351	1.381	85.927	8.485	2.975	.524	.709	42.296
112	1407	1.383	85.918	8.493	2.979	.521	.706	42.295
113	1422	1.383	85.930	8.475	2.981	.521	.710	42.294
114	1437	1.382	85.910	8.493	2.983	.522	.710	42.302
115	1452	1.383	85.915	8.488	2.980	.525	.709	42.300
116	1507	1.381	85.927	8.482	2.983	.522	.705	42.295
117	1522	1.384	85.935	8.476	2.974	.524	.707	42.290
118	1538	1.380	85.936	8.478	2.976	.522	.709	42.293
119	1553	1.379	85.923	8.489	2.978	.520	.711	42.298
120	1608	1.383	85.935	8.473	2.975	.523	.711	42.293
121	1623	1.378	85.943	8.483	2.973	.520	.703	42.287
122	1639	1.378	85.923	8.489	2.977	.524	.709	42.300
201	1102	1.382	85.907	8.496	2.985	.522	.709	42.303
202	1117	1.381	85.938	8.474	2.979	.522	.706	42.291
203	1133	1.382	85.922	8.487	2.975	.521	.713	42.297
204	1148	1.385	85.900	8.507	2.978	.520	.710	42.300
205	1204	1.378	85.967	8.463	2.966	.520	.706	42.280
206	1219	1.387	85.910	8.500	2.974	.519	.710	42.294
207	1234	1.386	85.911	8.490	2.984	.522	.707	42.298
208	1350	1.386	85.902	8.503	2.980	.521	.708	42.299
209	1306	1.385	85.901	8.498	2.982	.520	.714	42.303
210	1331	1.386	85.905	8.508	2.977	.521	.703	42.294
211	1336	1.383	85.905	8.500	2.984	.520	.708	42.301
212	1352	1.384	85.892	8.507	2.989	.521	.707	42.305
213	1407	1.385	85.913	8.490	2.982	.520	.710	42.297
214	1422	1.384	85.913	8.488	2.982	.523	.710	42.300
215	1438	1.384	85.885	8.513	2.981	.524	.713	42.310
216	1453	1.385	85.906	8.500	2.979	.524	.707	42.299
217	1508	1.385	85.887	8.500	2.997	.524	.708	42.310
218	1524	1.383	85.886	8.520	2.980	.521	.711	42.307
219	1539	1.384	85.911	8.498	2.978	.523	.706	42.297
220	1554	1.384	85.902	8.499	2.986	.523	.706	42.302
221	1609	1.383	85.890	8.514	2.982	.523	.708	42.306
222	1624	1.384	85.908	8.494	2.982	.522	.710	42.301

7.1.3. Evaluation of gas chromatograph column configuration and digital integrator.

Table 7.1.5 lists the data used in the evaluation of column configuration A; table 7.1.6 gives comparable data for column configuration B. Table 7.1.7 compares the two integrators. All of these tests used prepared mixture H.

Table 7.1.5. Column configuration A evaluation data.

Date	Composition, mol %								Heating Value MJ/m ³
	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	
8/1/77	.603	90.123	6.527	2.197	.291	.238	.009	.013	41.030
8/1/77	.598	90.108	6.540	2.203	.291	.239	.010	.011	41.039
8/1/77	.599	90.112	6.537	2.200	.292	.239	.010	.012	41.037
8/1/77	.593	90.108	6.543	2.204	.290	.239	.010	.012	41.043
8/1/77	.599	90.115	6.541	2.194	.291	.238	.011	.011	41.034
8/1/77	.597	90.104	6.543	2.203	.292	.239	.010	.012	41.041
8/1/77	.597	90.118	6.545	2.191	.292	.236	.010	.012	41.033
8/1/77	.600	90.109	6.544	2.196	.291	.238	.010	.012	41.036
8/1/77	.594	90.109	6.549	2.195	.291	.239	.010	.013	41.041
8/1/77	.599	90.102	6.547	2.198	.291	.239	.010	.013	41.040
8/1/77	.597	90.117	6.544	2.192	.291	.238	.010	.012	41.034
8/1/77	.593	90.119	6.542	2.195	.291	.240	.010	.011	41.037
8/2/77	.593	90.114	6.540	2.201	.292	.239	.009	.012	41.041
8/2/77	.602	90.104	6.536	2.207	.291	.238	.010	.011	41.038
8/2/77	.601	90.119	6.536	2.193	.291	.239	.011	.011	41.030
8/2/77	.601	90.115	6.539	2.193	.291	.238	.010	.012	41.032
8/2/77	.602	90.101	6.542	2.204	.291	.238	.010	.012	41.039
8/2/77	.602	90.114	6.539	2.195	.291	.238	.010	.012	41.032
8/2/77	.603	90.129	6.532	2.192	.288	.235	.009	.011	41.022
8/2/77	.610	90.106	6.534	2.193	.291	.238	.013	.013	41.033
8/2/77	.606	90.099	6.542	2.202	.291	.238	.010	.012	41.036
8/2/77	.604	90.102	6.542	2.201	.290	.238	.010	.012	41.036
8/2/77	.602	90.098	6.546	2.200	.291	.240	.010	.013	41.040
8/2/77	.611	90.030	6.609	2.201	.291	.238	.010	.010	41.050
8/2/77	.615	90.090	6.547	2.196	.291	.240	.010	.011	41.032
8/2/77	.621	90.111	6.548	2.167	.291	.241	.010	.012	41.014
8/2/77	.604	90.138	6.545	2.165	.290	.237	.010	.011	41.014
8/2/77	.606	90.079	6.558	2.204	.291	.240	.010	.012	41.042

Table 7.1.6. Column configuration B evaluation data.

Date	Composition, mol %								Heating Value MJ/m ³
	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	
9/17/77	.630	90.108	6.478	2.190	.290	.282	.013	.010	41.039
9/17/77	.585	90.000	6.602	2.214	.293	.286	.009	.012	41.108
9/17/77	.580	90.098	6.530	2.196	.291	.283	.008	.012	41.076
9/17/77	.583	90.023	6.586	2.212	.292	.284	.008	.012	41.101
9/19/77	.592	90.080	6.534	2.199	.291	.283	.010	.012	41.075
9/19/77	.594	90.046	6.556	2.207	.292	.284	.010	.011	41.086
9/19/77	.610	90.082	6.520	2.192	.291	.283	.010	.011	41.060
9/19/77	.591	90.034	6.572	2.206	.292	.284	.010	.012	41.092
9/19/77	.595	90.031	6.560	2.214	.292	.285	.010	.012	41.093
9/19/77	.591	90.059	6.553	2.201	.290	.284	.010	.013	41.083
9/19/77	.592	90.052	6.555	2.203	.292	.283	.010	.013	41.085

Table 7.1.7. Data for the comparison between integrators A and B.

Integrator	Composition, mol %								Heating Value MJ/m ³
	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	
A	.598	90.107	6.535	2.212	.291	.236	.011	.010	41.040
B	.595	90.107	6.544	2.204	.292	.240	.010	.010	41.041
A	.607	90.109	6.539	2.197	.291	.236	.010	.011	41.029
B	.610	90.090	6.544	2.205	.290	.238	.011	.012	41.037
A	.599	90.110	6.539	2.201	.291	.239	.010	.011	41.037
B	.592	90.122	6.533	2.199	.292	.239	.010	.012	41.039
A	.620	90.098	6.536	2.199	.291	.236	.010	.011	41.024
B	.621	90.099	6.531	2.200	.290	.238	.009	.012	41.024
A	.601	90.105	6.539	2.206	.292	.235	.011	.011	41.037
B	.597	90.118	6.533	2.201	.290	.239	.012	.012	41.038
A	.619	90.090	6.542	2.199	.292	.238	.010	.011	41.029
B	.620	90.077	6.546	2.201	.292	.241	.012	.011	41.037
A	.607	90.108	6.534	2.202	.291	.237	.010	.011	41.032
B	.605	90.121	6.524	2.201	.289	.239	.011	.010	41.029
A	.604	90.099	6.538	2.206	.292	.237	.013	.010	41.039
B	.616	90.078	6.542	2.206	.289	.241	.014	.014	41.042
A	.601	90.108	6.533	2.208	.292	.237	.010	.011	41.038
B	.605	90.107	6.532	2.208	.290	.237	.009	.011	41.033
A	.602	90.104	6.542	2.202	.292	.237	.010	.012	41.038
B	.610	90.099	6.539	2.202	.290	.236	.011	.013	41.034
A	.603	90.098	6.541	2.205	.293	.238	.010	.012	41.040
B	.609	90.108	6.523	2.204	.289	.240	.013	.014	41.036

7.1.4. Determination of the effect of operating variables on gas chromatograph analysis accuracy and precision.

Table 7.1.8 lists the data for various sample pressures; table 7.1.9 lists the data for different carrier gas flow rates. Prepared mixture H was used throughout these tests.

Table 7.1.8. Data for determining the effect of sample pressure on gas chromatograph analysis accuracy and precision.

Sample Pressure kPa	Integrator	Composition, mol %								Heating Value MJ/m ³
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	
94	A	.594	90.073	6.534	2.204	.291	.283	.010	.011	41.076
94	B	.597	90.062	6.543	2.201	.292	.284	.010	.011	41.078
94	A	.598	90.065	6.529	2.214	.290	.282	.010	.011	41.078
94	B	.605	90.066	6.535	2.201	.289	.283	.011	.010	41.069
94	A	.599	90.069	6.537	2.200	.291	.284	.010	.011	41.074
94	B	.594	90.071	6.534	2.201	.293	.284	.009	.013	41.079
94	A	.595	90.070	6.533	2.208	.290	.284	.011	.011	41.077
94	B	.604	90.065	6.540	2.198	.289	.283	.008	.013	41.069
94	A	.595	90.066	6.539	2.202	.290	.285	.010	.012	41.079
94	B	.596	90.057	6.554	2.194	.293	.286	.009	.011	41.078
94	A	.592	90.067	6.538	2.206	.291	.283	.010	.013	41.081
94	B	.589	90.067	6.547	2.199	.291	.285	.008	.013	41.081
145	A	.605	89.930	6.621	2.240	.295	.287	.010	.011	41.124
145	B	.601	89.951	6.624	2.225	.293	.287	.009	.010	41.113
145	A	.605	89.953	6.619	2.219	.295	.287	.010	.011	41.112
145	B	.605	89.949	6.617	2.225	.295	.290	.009	.011	41.114
145	A	.606	89.940	6.620	2.231	.295	.288	.010	.011	41.118
145	B	.601	89.942	6.629	2.225	.294	.288	.009	.012	41.119
145	A	.608	89.943	6.619	2.225	.296	.288	.010	.012	41.115
145	B	.606	89.940	6.623	2.226	.293	.289	.010	.013	41.118
145	A	.605	89.944	6.624	2.222	.295	.288	.010	.011	41.115
145	B	.602	89.942	6.628	2.221	.295	.290	.010	.013	41.119
59	A	.587	90.159	6.480	2.189	.286	.279	.010	.011	41.046
59	B	.618	90.121	6.489	2.178	.286	.284	.011	.013	41.039
59	A	.592	90.164	6.473	2.185	.286	.279	.011	.010	41.041
59	B	.607	90.142	6.483	2.178	.290	.280	.011	.011	41.039
59	A	.590	90.173	6.471	2.177	.288	.280	.009	.012	41.039
59	B	.607	90.147	6.482	2.177	.290	.279	.009	.010	41.034
59	A	.592	90.169	6.467	2.183	.287	.282	.009	.011	41.040
59	B	.589	90.162	6.480	2.173	.291	.284	.007	.014	41.046
59	A	.601	90.152	6.470	2.190	.287	.279	.010	.011	41.040
59	B	.616	90.139	6.476	2.180	.289	.281	.009	.011	41.033

Table 7.1.9. Data for determining the effect of carrier gas flow rate on gas chromatograph analysis accuracy and precision.

Flow Rate scm ³ /min	Integrator	Composition, mol %								Heating Value MJ/m ³
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	
26.5	A	.599	90.068	6.533	2.202	.292	.283	.010	.012	41.075
26.5	B	.603	90.065	6.539	2.200	.290	.283	.009	.010	41.069
26.5	A	.597	90.071	6.537	2.198	.291	.283	.010	.012	41.074
26.5	B	.594	90.073	6.532	2.203	.291	.285	.010	.012	41.078
26.5	A	.599	90.068	6.537	2.200	.291	.284	.010	.011	41.074
26.5	B	.599	90.057	6.544	2.201	.292	.283	.011	.012	41.078
26.5	A	.598	90.056	6.539	2.209	.292	.284	.010	.011	41.081
26.5	B	.599	90.065	6.539	2.201	.291	.284	.009	.012	41.074
26.5	A	.600	90.072	6.540	2.191	.293	.283	.010	.012	41.070
26.5	B	.595	90.071	6.541	2.198	.291	.284	.009	.011	41.074
24.2	A	.606	90.040	6.531	2.227	.293	.284	.010	.008	41.083
24.2	B	.525	90.114	6.547	2.214	.294	.287	.010	.009	41.115
24.2	A	.607	90.065	6.517	2.218	.292	.283	.010	.008	41.072
24.2	B	.522	90.153	6.533	2.198	.291	.284	.010	.009	41.097
24.2	A	.610	90.045	6.528	2.224	.293	.283	.010	.008	41.077
24.2	B	.518	90.127	6.552	2.206	.293	.286	.009	.008	41.111
24.2	A	.610	90.043	6.535	2.221	.292	.283	.010	.007	41.076
24.2	B	.514	90.138	6.551	2.205	.291	.285	.009	.007	41.108
24.2	A	.609	90.049	6.534	2.216	.292	.283	.010	.007	41.073
24.2	B	.511	90.141	6.551	2.205	.291	.284	.010	.007	41.109
27.6	A	.607	90.062	6.537	2.201	.292	.279	.010	.012	41.069
27.6	B	.614	89.921	6.630	2.230	.297	.289	.009	.011	41.119
27.6	A	.608	90.053	6.537	2.204	.293	.284	.010	.011	41.074
27.6	B	.612	89.924	6.632	2.229	.296	.289	.009	.009	41.118
27.6	A	.604	90.109	6.523	2.168	.291	.283	.010	.011	41.049
27.6	B	.617	89.920	6.630	2.224	.297	.288	.010	.012	41.117
27.6	A	.608	90.093	6.544	2.157	.293	.284	.011	.011	41.050
27.6	B	.616	89.875	6.662	2.240	.298	.288	.009	.011	41.134
27.6	A	.607	90.046	6.545	2.203	.292	.285	.010	.011	41.077
27.6	B	.615	89.883	6.655	2.234	.297	.292	.011	.012	41.134

APPENDIX 7.2. RECOMMENDED CRITERIA FOR SELECTING A GAS CHROMATOGRAPH
FOR USE IN ANALYZING VAPORIZED LNG SAMPLES

Based on our experiences during this project, we recommend the following features be incorporated into any gas chromatographs used for analyzing vaporized LNG samples:

- a) automatic valve switching,
- b) hot-wire filament detector with a thermal protect device to minimize the risk of oxidizing the filaments; the detector cavity should have minimal gas volume,
- c) a separate electrical input line for the detector power supply,
- d) carrier gas flow control by pressure instead of mass flow, and
- e) valve and column ovens which are accesible for leak checking all fittings.

As a precaution, we recommend that the thermal conductivity detector power input be connected to a constant voltage transformer.

For the digital integrator we recommend that the "memory protect" feature be used; also, it is recommended that a constant voltage transformer be used with the digital integrator.

APPENDIX 7.3. METHOD FOR COMPUTING THE UNCERTAINTY OF A GAS ANALYSIS AND A METHOD FOR REJECTING A GIVEN ANALYSIS

This appendix suggests useful statistical techniques for computing the uncertainty in composition measurement; also, it describes a simple procedure for rejecting a given analysis from a set of replicate analyses of the same sample. Examples are given for both methods.

7.3.1. Computing the composition uncertainty of a given sample.

As mentioned in section 2.2., a logical way to estimate the error in a measurement process is to treat the error as composed of two parts -- accuracy and precision. By following the recommendations of section 4.6.1, the only source of bias in a gas chromatograph analysis is the error in the calibration gas composition. The total uncertainty ($\pm D$) (and how they estimated it) of each component's composition should be given by the supplier of the calibration gas. If possible, the composition should be verified by another gas analysis facility.

The size of the random error (i.e., standard deviation) will be unique to each gas chromatograph-integrator system; however, comparable systems should have comparable standard deviations. Test results showed that the standard deviation for calibration and analyses can be assumed equal. If we make N_C calibration runs before analyzing a gas sample N_A times, the value and total uncertainty of a given component (or derived property such as heating value) will be

$$\bar{x} \pm (D + s \cdot t \sqrt{1/N_A + 1/N_C}) \quad (7.3.1)$$

where $\bar{x}$ is the average value (i.e., $\bar{x} = \frac{1}{N_A} \sum_{i=1}^{N_A} x_i$), s is an estimate, based on replicate measurements, of the standard deviation and t the Student's t statistic for the desired confidence interval (e.g., 99 percent). There are two choices for the value of s -- one is to compute s from measurements on the given sample, i.e.

$$s = \sqrt{\frac{1}{N_A} \sum_{i=1}^{N_A} (x_i - \bar{x})^2 / (N_A - 1)}. \quad (7.3.2)$$

The second, and preferred, choice is to use the composite standard deviation, s_p , which is based on all previous sets of analyses. Since s_p is computed from many analyses it more closely represents the true standard deviation of the measurement process. The composite, or pooled, standard deviation is given by

$$s_p = \sqrt{\frac{\sum_{j=1}^M (N_{Aj} - 1) s_j^2}{\sum_{j=1}^M (N_{Aj} - 1)}} \quad (7.3.3)$$

where N_{Aj} is the number of replicate analyses taken on the j th sample, s_j is the estimated standard deviation for that set (computed using 7.3.2) and M is the total number of samples analyzed.

The Student's t statistic used will depend upon the chosen confidence interval and the chosen standard deviation (s or s_p).

This approach could be applied to the sampling system if several samples could be taken during a period when the LNG composition is constant.

Example 1

The results of four replicate analyses of a sample give the following methane contents: 90.080, 90.032, 90.080 and 90.070 mol percent. Prior to these analyses, there were three calibration runs in which the response factors for each component were averaged. The stated uncertainty in the methane content of the calibration gas is ± 0.003 mol percent.

The average methane value is 90.066 mol percent and the estimated standard deviation, s , is 0.023. Using these values, the Student's t statistic for three degrees of freedom (i.e., $N_A - 1$) and a 99 percent confidence interval from table 7.3.1 of 5.841, we obtain the total uncertainty in the methane content:

$$\begin{aligned} & \pm (D + s \cdot t \sqrt{1/N_A + 1/N_C}) \\ & \pm (0.003 + 0.023 \cdot 5.841 \sqrt{1/4 + 1/3}) \\ & \pm 0.106 \text{ mol } \%. \end{aligned}$$

However, prior to this analysis, a sufficient number of analyses were made so that $\sum_j (N_{Aj} - 1) = 52$ (which is the number of degrees of freedom), and the composite standard deviation, s_p , is 0.020. Based on 52 degrees of freedom, t is approximately 2.68 and the total uncertainty becomes

$$\pm (0.003 + 0.020 \cdot 2.68 \sqrt{1/4 + 1/3}) = \pm 0.044 \text{ mol } \%.$$

This value is 60 percent smaller than the other; also, it is more representative of the expected uncertainty.

The size of the confidence level depends upon the number of analyses; however, the width decreases as the square root of $1/N_A$. This makes it unattractive to make more than three or four analyses. If there had been ten analyses instead of four in the above example the confidence interval would be ± 0.038 ; this is only 13 percent smaller than when using four analyses.

The value of s_p can be updated using the above data and equation 7.3.3, i.e.,

$$s_p = \sqrt{(52 \cdot (0.020)^2 + 3(0.023)^2) / (52 + 3)} = 0.0202$$

which is a negligible change in this case.

Table 7.3.1. Values of the Student's t distribution used to calculate confidence intervals for the mean [5].

Degrees of Freedom	Student's t Distribution Confidence Interval	
	95%	99%
1	12.706	63.657
2	4.303	9.925
3	3.182	5.841
4	2.776	4.604
5	2.571	4.032
6	2.447	3.707
7	2.365	3.499
8	2.306	3.355
9	2.262	3.250
10	2.228	3.169
11	2.201	3.106
12	2.179	3.055
13	2.160	3.012
14	2.145	2.977
15	2.131	2.947
16	2.120	2.921
17	2.110	2.898
18	2.101	2.878
19	2.093	2.861
20	2.086	2.845
21	2.080	2.831
22	2.074	2.819
23	2.069	2.807
24	2.064	2.797
25	2.060	2.787
26	2.056	2.779
27	2.052	2.771
28	2.048	2.763
29	2.045	2.756
30	2.042	2.750
40	2.021	2.704
60	2.000	2.660
120	1.980	2.617
∞	1.960	2.576

7.3.2. Methods of rejecting an analysis.

To reject a given analysis we need to run two or more analyses and compute the difference, R , between the maximum and minimum measured values. One of these two points can be rejected if

$$R = x_{\max} - x_{\min} > A \cdot s_p \quad (7.3.4)$$

where A is a function of the number of analyses, N_A (ref. [5], p. 17-3). Table 7.3.2 lists the values of A . Using this criterion, only one out of one hundred rejected analyses will be rejected when it should not have been.

Table 7.3.2. Values of A as a function of N_A .

N_A	A
2	3.64
3	4.12
4	4.40
5	4.60
6	4.76
7	4.88
8	4.99
9	5.08
10	5.16

Example 2

Three replicate analyses are run and the measured methane contents are 90.132, 90.021 and 90.032 mol percent. From previous analyses we know that s_p is 0.020. Can the first value be rejected?

Using equation 7.3.4,

$$R = 90.132 - 90.021 > \frac{4.12 \cdot 0.020}{0.111} > 0.082.$$

Therefore, the first point should be rejected. However, if the first value had been 90.103 mol percent, it would not be rejected using this procedure.

APPENDIX 7.4 SAMPLING DATA

This appendix lists all of the measured operating variables in the sampling tests according to run number. The sample analysis along with the computed liquid density are listed after operating variables for each test. All compositions were measured using integrator A unless stated otherwise.

7.4.1. Laboratory Test Data

In table 7.4.1 "Mix" refers to samples of the mixture present in the apparatus during the given test, e.g., 1; analyses of the corresponding gas cylinder will have a Mix number greater than 100, e.g., 101. Mix 100 represents analyses of the calibration gas (see table 4.2, mixture E). The listed heating values are ideal gas values computed using the values given in table 2.2; to convert from MJ/m³ to Btu/scf multiply by 26.845.

Table 7.4.1. Laboratory Test Data Probe Test 1 (runs 20 through 32 were not used in any statistical evaluation).

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KFA	PROBE	VAPORIZER POWER, WATTS
1	12/22	113.0	.099	3.41	2.0	25.	S	7.6
2	12/22	113.0	.099	3.41	2.0	25.	R	7.0
3	12/22	113.0	.099	3.44	2.0	25.	P	7.0
4	12/22	113.0	.100	3.48	6.0	25.	P	64.0
5	12/23	113.0	.100	3.44	6.0	25.	S	70.1
6	12/23	113.0	.101	3.41	6.0	26.	R	70.6
7	12/23	113.0	.100	3.44	2.0	27.	P	6.8
8	12/23	113.0	.100	3.48	2.0	19.	P	12.6
9	12/23	113.0	.101	3.44	2.0	12.	P	2.5
10	12/23	113.0	.103	5.79	2.0	12.	P	4.0
11	12/23	113.0	.102	5.90	2.0	13.	R	4.0
12	12/27	113.0	.102	1.93	2.0	25.	R	8.9
13	12/27	113.0	.102	1.89	2.0	25.	S	8.9
14	12/27	113.0	.102	1.89	2.0	25.	P	8.9
15	12/27	113.0	.102	1.89	6.0	25.	P	67.7
16	12/27	113.0	.102	1.85	6.0	25.	S	64.4
17	12/27	113.0	.103	1.89	6.0	25.	R	67.7
18	12/28	120.0	.173	3.67	4.6	25.	R	44.6
19	12/28	120.0	.173	3.79	4.6	25.	P	45.9
20	12/28	120.0	.173	3.79	4.6	25.	S	47.3
21	12/28	113.0	.124	3.71	2.2	25.	S	10.1
22	12/28	113.0	.125	3.75	2.2	25.	R	11.6
23	12/28	113.0	.125	3.75	2.2	25.	P	11.4
24	12/29	113.0	.126	3.75	2.2	25.	S	10.8
25	12/29	113.0	.126	3.79	2.2	25.	R	10.8
26	12/29	113.0	.126	3.79	2.2	25.	P	13.1
27	12/29	113.0	.126	3.75	6.7	24.	P	68.5
28	12/29	113.1	.124	3.79	6.7	26.	R	69.7
29	12/29	113.0	.127	3.79	6.7	25.	S	69.7
30	1/03	113.0	.251	3.79	3.1	25.	S	31.4
31	1/03	113.0	.252	3.82	3.2	25.	P	33.4
32	1/03	113.0	.253	3.79	3.2	25.	R	33.1

Table 7.4.1. Probe Test 1 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
1	1307	1	0.000	85.648	11.680	1.401	.541	.730	42.982	.4691
2	1346	1	0.000	85.667	11.640	1.399	.554	.740	42.989	.4691
2	1404	1	0.000	85.692	11.620	1.391	.553	.744	42.981	.4690
2	1426	101	0.000	85.619	11.720	1.386	.539	.736	42.988	.4691
3	1451	1	0.000	85.557	11.760	1.399	.544	.740	43.015	.4694
3	1507	1	0.000	85.533	11.760	1.403	.552	.751	43.033	.4695
3	1524	1	0.000	85.546	11.759	1.404	.548	.743	43.023	.4694
4	1547	1	0.000	85.689	11.660	1.381	.544	.726	42.964	.4689
4	1602	1	0.000	85.691	11.650	1.382	.543	.734	42.968	.4689
5	1012	1	0.000	85.611	11.699	1.401	.547	.742	43.003	.4692
5	1031	101	0.000	85.610	11.710	1.391	.555	.734	43.000	.4692
6	1059	1	0.000	85.675	11.649	1.390	.550	.736	42.980	.4690
6	1115	1	0.000	85.649	11.680	1.384	.548	.739	42.986	.4691
7	1251	1	0.000	85.723	11.619	1.378	.547	.733	42.959	.4689
7	1308	1	0.000	85.707	11.640	1.375	.546	.730	42.962	.4689
8	1347	1	0.000	85.663	11.650	1.394	.551	.741	42.988	.4691
8	1404	1	0.000	85.693	11.630	1.395	.549	.733	42.974	.4690
8	1422	101	0.000	85.608	11.720	1.394	.547	.731	42.995	.4692
9	1447	1	0.000	85.723	11.610	1.380	.548	.739	42.964	.4689
9	1504	1	0.000	85.734	11.609	1.371	.549	.737	42.957	.4688
10	1531	1	0.000	85.503	11.730	1.417	.574	.775	43.071	.4698
10	1549	1	0.000	85.517	11.730	1.415	.571	.768	43.061	.4697
11	1607	1	0.000	85.679	11.640	1.388	.548	.745	42.982	.4691
11	1623	1	0.000	85.695	11.641	1.380	.547	.737	42.970	.4690
11	1641	101	0.000	85.555	11.741	1.405	.554	.745	43.025	.4694
12	1019	1	0.000	85.672	11.675	1.377	.540	.736	42.971	.4690
12	1038	1	0.000	85.691	11.637	1.384	.546	.742	42.974	.4690
12	1057	1	0.000	85.674	11.664	1.379	.544	.739	42.975	.4690
12	956	101	0.000	85.591	11.739	1.394	.543	.734	43.000	.4692
13	1144	1	0.000	85.683	11.649	1.386	.544	.738	42.974	.4690
13	1203	1	0.000	85.728	11.630	1.380	.537	.725	42.948	.4688
14	1235	1	0.000	85.664	11.621	1.409	.557	.749	42.999	.4692
14	1252	1	0.000	85.666	11.621	1.407	.553	.753	42.998	.4692
15	1336	1	0.000	85.648	11.689	1.393	.540	.730	42.979	.4691
15	1354	1	0.000	85.649	11.685	1.397	.539	.730	42.980	.4691
15	1410	1	0.000	85.643	11.674	1.400	.543	.740	42.990	.4691
16	1605	1	0.000	85.764	11.611	1.371	.534	.720	42.931	.4686
16	1621	1	0.000	85.703	11.659	1.378	.537	.723	42.954	.4688
16	1546	101	0.000	85.672	11.699	1.375	.536	.718	42.958	.4689
17	1642	1	0.000	85.637	11.670	1.381	.536	.726	42.960	.4689
17	1658	1	0.000	85.696	11.669	1.378	.534	.723	42.954	.4688
18	1002	1	0.000	85.713	11.660	1.377	.529	.720	42.945	.4589
18	1018	1	0.000	85.707	11.667	1.384	.527	.715	42.944	.4589
18	1034	1	0.000	85.723	11.670	1.373	.525	.719	42.932	.4588
19	1112	1	0.000	85.565	11.719	1.420	.548	.748	43.025	.4596
19	1130	1	0.000	85.511	11.759	1.420	.553	.758	43.048	.4598
19	1147	1	0.000	85.487	11.785	1.419	.550	.759	43.054	.4598
19	1203	1	0.000	85.503	11.771	1.425	.550	.751	43.047	.4598
20	1251	1	0.000	85.713	11.660	1.380	.534	.712	42.944	.4589
20	1308	1	0.000	85.727	11.651	1.382	.524	.716	42.937	.4588

Table 7.4.1. Probe Test 1 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
20	1224	101	0.003	85.635	11.721	1.390	.527	.727	42.973	.4591
21	1521	2	.543	85.181	11.619	1.385	.540	.733	42.752	.4703
21	1555	2	.542	85.200	11.610	1.383	.535	.730	42.742	.4703
22	1613	2	.558	84.947	11.753	1.426	.557	.762	42.845	.4712
22	1645	2	.542	84.952	11.749	1.436	.557	.764	42.858	.4712
23	1708	2	.544	85.162	11.649	1.380	.531	.734	42.750	.4703
23	1724	2	.544	85.147	11.660	1.387	.535	.728	42.756	.4704
24	1043	2	.546	85.231	11.580	1.384	.535	.724	42.728	.4702
24	1059	2	.546	85.223	11.590	1.378	.532	.730	42.730	.4702
25	1136	2	.547	85.289	11.520	1.381	.537	.726	42.712	.4700
25	1152	2	.548	85.279	11.538	1.370	.538	.727	42.712	.4700
25	1213	102	.026	85.877	11.510	1.346	.538	.704	42.868	.4683
26	1236	2	.548	85.095	11.639	1.415	.555	.748	42.798	.4707
26	1251	2	.548	85.045	11.649	1.442	.563	.753	42.827	.4710
26	1309	2	.546	85.063	11.639	1.434	.554	.764	42.822	.4709
27	1341	2	.548	85.225	11.579	1.380	.538	.730	42.732	.4702
27	1357	2	.547	85.234	11.579	1.376	.537	.727	42.727	.4701
27	1420	100	1.384	85.887	8.505	2.989	.523	.712	42.423	.4717
28	1446	2	.549	85.263	11.569	1.372	.527	.720	42.706	.4699
28	1503	2	.549	85.269	11.560	1.377	.532	.713	42.705	.4698
29	1542	2	.548	85.240	11.580	1.379	.533	.720	42.719	.4701
29	1557	2	.547	85.265	11.569	1.369	.529	.721	42.708	.4700
29	1525	101	0.000	85.731	11.660	1.371	.535	.703	42.932	.4687
30	1000	3	4.339	81.962	11.160	1.324	.520	.695	41.104	.4789
30	1026	3	4.339	81.968	11.140	1.334	.519	.701	41.107	.4789
31	1113	3	4.330	81.891	11.200	1.353	.523	.703	41.144	.4792
31	1131	3	4.335	81.908	11.180	1.350	.525	.702	41.135	.4791
31	1159	101	0.000	85.615	11.719	1.396	.546	.724	42.989	.4693
32	1225	3	4.333	82.007	11.130	1.324	.513	.694	41.090	.4788
32	1242	3	4.340	81.993	11.139	1.324	.515	.690	41.089	.4788

Table 7.4.1. Probe Test 2

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	PROBE	VAPORIZER POWER, WATTS
33	1/03	113.0	.147	3.75	2.4	26.	R	16.2
34	1/03	113.0	.148	3.71	2.4	25.	P	17.4
35	1/03	113.0	.148	3.79	2.4	26.	S	18.9
36	1/03	113.0	.148	3.79	2.4	25.	R	16.0
37	1/03	113.0	.148	3.79	2.4	25.	S	16.0
38	1/03	113.0	.148	3.79	2.4	25.	P	16.0
39	1/04	113.0	.142	3.82	7.1	25.	S	94.1
40	1/04	113.0	.149	3.79	7.3	25.	R	78.3
41	1/04	113.0	.147	3.79	2.4	25.	P	73.5
42	1/04	113.0	.147	3.79	7.2	25.	R	77.4
43	1/04	113.0	.147	3.75	7.2	25.	S	77.9
44	1/04	113.0	.147	3.79	7.2	25.	P	77.9
45	1/04	113.0	.150	3.75	2.4	25.	P	18.9
46	1/04	113.0	.150	3.79	2.4	25.	S	36.5
47	1/04	113.0	.149	3.75	2.4	25.	R	13.7
48	1/04	113.0	.150	3.75	2.4	25.	R	18.7
49	1/05	113.0	.148	3.75	2.4	25.	R	19.1
50	1/05	113.0	.148	3.79	2.4	25.	S	19.4
51	1/05	113.0	.149	3.75	2.4	25.	P	19.4
52	1/05	113.0	.149	3.82	7.2	25.	S	71.4
53	1/05	113.0	.150	3.79	7.1	26.	P	66.0
54	1/05	113.0	.150	3.79	7.0	25.	R	74.8
55	1/05	113.0	.152	3.79	5.8	26.	S	58.5
56	1/05	113.0	.150	3.79	5.5	26.	P	54.8
57	1/05	113.0	.155	3.63	5.6	24.	R	52.2

Table 7.4.1. Probe Test 2 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
33	1323	4	1.340	83.913	11.970	1.450	.569	.757	42.632	.4741
33	1340	4	1.341	83.927	11.960	1.450	.566	.756	42.625	.4740
34	1426	4	1.342	83.907	11.970	1.445	.570	.766	42.637	.4741
35	1448	4	1.339	83.960	11.950	1.419	.570	.762	42.614	.4739
35	1505	4	1.338	83.985	11.939	1.429	.552	.757	42.598	.4738
36	1548	4	1.341	83.885	11.959	1.458	.577	.780	42.659	.4743
36	1603	4	1.335	83.919	11.940	1.449	.581	.776	42.651	.4742
36	1620	101	0.000	85.713	11.674	1.379	.534	.700	42.937	.4688
37	1639	4	1.338	83.777	12.040	1.480	.581	.785	42.703	.4746
37	1655	4	1.336	83.779	12.030	1.490	.587	.778	42.706	.4746
38	1727	4	1.333	83.798	12.000	1.491	.589	.784	42.704	.4746
38	826	4	1.336	83.753	12.020	1.493	.589	.809	42.733	.4748
39	1006	4	1.341	83.946	11.979	1.425	.555	.754	42.606	.4739
39	1022	4	1.340	83.946	11.979	1.426	.555	.754	42.607	.4739
39	856	101	0.000	85.605	11.729	1.392	.536	.738	42.994	.4692
40	1039	4	1.341	83.961	11.970	1.425	.551	.752	42.598	.4738
40	1055	4	1.350	83.934	11.969	1.430	.559	.758	42.609	.4739
40	1111	4	1.338	83.952	11.979	1.417	.555	.759	42.606	.4739
41	1201	4	1.339	84.090	11.870	1.403	.548	.750	42.554	.4734
41	1217	4	1.338	84.084	11.879	1.408	.546	.745	42.554	.4734
42	1250	4	1.340	83.952	11.969	1.438	.549	.752	42.604	.4738
43	1319	4	1.354	83.798	12.080	1.437	.560	.771	42.655	.4744
43	1336	4	1.343	83.964	11.959	1.424	.553	.757	42.600	.4738
44	1424	4	1.340	83.947	11.980	1.425	.555	.754	42.606	.4739
44	1440	4	1.340	83.956	11.969	1.429	.552	.754	42.603	.4738
45	1459	4	1.338	83.898	11.990	1.440	.563	.771	42.640	.4741
45	1515	4	1.351	83.887	11.990	1.450	.561	.762	42.631	.4741
46	1533	4	1.336	83.984	11.961	1.425	.547	.747	42.590	.4737
46	1550	4	1.337	83.962	11.980	1.423	.553	.745	42.597	.4738
47	1609	4	1.338	83.942	11.980	1.424	.557	.759	42.613	.4739
47	1625	4	1.340	83.905	11.989	1.440	.560	.766	42.632	.4741
48	1644	4	1.338	83.953	11.950	1.424	.561	.774	42.620	.4740
48	1700	4	1.341	83.939	11.968	1.429	.557	.766	42.616	.4739
48	1716	101	0.000	85.649	11.702	1.377	.543	.729	42.976	.4691
49	911	4	1.340	83.965	11.961	1.423	.563	.748	42.601	.4738
49	944	4	1.331	83.965	11.969	1.419	.558	.758	42.609	.4738
50	1008	4	1.337	83.997	11.940	1.421	.555	.751	42.591	.4737
50	1024	4	1.340	83.993	11.930	1.431	.555	.751	42.593	.4737
51	1044	4	1.339	83.862	11.999	1.456	.570	.775	42.660	.4743
51	1059	4	1.352	83.853	11.999	1.459	.571	.766	42.650	.4743
51	1120	101	0.000	85.617	11.730	1.389	.538	.726	42.984	.4691
52	1143	4	1.335	83.967	11.970	1.424	.557	.748	42.601	.4738
52	1159	4	1.332	83.969	11.980	1.418	.559	.742	42.598	.4738
53	1221	4	1.337	83.974	11.971	1.429	.552	.737	42.590	.4737
53	1255	4	1.334	83.984	11.961	1.429	.549	.743	42.591	.4737
54	1321	4	1.340	83.954	11.969	1.427	.555	.755	42.605	.4739
54	1337	4	1.340	83.954	11.969	1.432	.555	.750	42.604	.4738
54	1350	101	0.000	85.634	11.709	1.400	.537	.720	42.978	.4691
55	1459	4	1.337	83.994	11.951	1.428	.550	.740	42.585	.4737
55	1517	4	1.339	83.957	11.970	1.437	.554	.743	42.600	.4738

Table 7.4.1. Probe Test 2 (continued)									Heating	Density
Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Value MJ/m ³	gm/cm ³
56	1538	4	1.341	83.969	11.970	1.420	.552	.748	42.593	.4738
56	1554	4	1.334	83.967	11.970	1.439	.551	.740	42.598	.4738
57	1611	4	1.343	83.963	11.950	1.432	.559	.753	42.603	.4739
57	1628	4	1.336	83.967	11.950	1.435	.561	.751	42.607	.4738
57	1640	101	0.000	85.607	11.720	1.404	.539	.730	42.993	.4692

Table 7.4.1. Probe Test 3

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	PROBE	VAPORIZER POWER, WATTS
58	1/31	111.0	.127	7.19	5.6	25.	S	57.0
59	1/31	111.0	.127	7.19	5.6	25.	R	57.0
60	1/31	111.0	.126	7.19	5.6	24.	P	60.8
61	1/31	111.0	.128	1.89	5.6	70.	R	58.9
62	1/31	111.0	.128	1.89	5.6	69.	S	64.0
63	1/31	111.0	.127	1.89	5.6	70.	P	69.3
64	1/31	111.1	.128	1.89	5.6	25.	P	64.0
65	1/31	111.0	.126	1.89	5.6	25.	S	64.0
66	1/31	111.0	.126	1.89	5.6	25.	R	64.4
67	1/31	120.0	.226	1.89	4.2	26.	R	46.2
68	1/31	120.0	.227	1.89	4.2	25.	P	45.6
69	1/31	120.0	.227	1.89	4.2	25.	S	45.6
70	2/01	120.0	.226	7.19	4.2	25.	S	53.7
71	2/01	120.0	.226	7.19	4.2	25.	R	49.7
72	2/01	120.0	.227	7.19	4.2	25.	P	47.6
73	2/01	120.0	.227	1.89	4.2	25.	R	50.4
74	2/01	120.0	.227	1.89	4.2	25.	S	50.4
75	2/01	120.0	.227	1.89	4.2	25.	P	48.3
76	2/01	120.0	.231	1.89	4.2	69.	R	40.6
77	2/01	120.0	.231	1.89	4.2	69.	S	43.6
78	2/01	120.0	.230	1.89	4.2	70.	P	41.6
79	2/02	111.0	.127	1.89	5.6	25.	S	63.6
80	2/02	111.0	.127	1.89	5.6	25.	P	47.6
81	2/02	111.0	.127	1.89	5.6	25.	R	60.5
82	2/02	111.0	.126	7.19	5.6	25.	P	62.8
83	2/02	111.0	.126	7.19	5.6	25.	S	66.4
84	2/02	111.0	.127	7.19	5.6	25.	R	67.2
85	2/02	111.0	.127	1.89	5.6	70.	P	66.4
86	2/02	120.0	.226	1.89	4.2	69.	S	51.5
87	2/02	120.0	.228	1.89	4.2	70.	R	49.3
88	2/03	120.0	.229	1.89	4.2	70.	S	38.4
89	2/03	120.0	.229	1.89	4.2	69.	R	41.6
90	2/03	120.0	.229	1.89	4.2	70.	P	41.6
91	2/03	120.0	.228	7.19	4.2	25.	S	41.0
92	2/03	120.0	.227	7.19	4.2	25.	P	41.0
93	2/03	120.0	.229	7.19	4.2	24.	R	41.0
94	2/03	111.0	.127	1.89	5.6	69.	R	63.2
95	2/03	111.0	.127	1.89	5.6	70.	S	52.2
96	2/03	111.0	.127	1.89	5.6	70.	P	63.2

Table 7.4.1. Probe Test 3 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
58	1058	5	1.219	84.565	9.510	3.524	.481	.701	43.028	.4788
58	1035	105	1.256	84.606	9.476	3.493	.479	.690	42.976	.4786
59	1124	5	1.220	84.546	9.535	3.514	.482	.704	43.032	.4789
60	1148	5	1.223	84.430	9.574	3.554	.494	.725	43.093	.4794
60	1205	5	1.222	84.447	9.552	3.558	.494	.728	43.091	.4793
61	1222	5	1.218	84.609	9.500	3.495	.481	.697	43.006	.4786
62	1247	5	1.219	84.563	9.527	3.505	.481	.705	43.026	.4788
62	1326	105	1.240	84.578	9.511	3.503	.479	.689	42.997	.4787
62	1311	105	1.237	84.565	9.526	3.500	.480	.692	43.004	.4787
63	1354	5	1.214	84.515	9.561	3.521	.484	.705	43.048	.4790
64	1419	5	1.218	84.585	9.519	3.505	.479	.694	43.013	.4785
65	1441	5	1.213	84.588	9.519	3.505	.479	.696	43.016	.4787
66	1504	5	1.215	84.547	9.525	3.523	.483	.707	43.040	.4789
67	1533	5	1.215	84.551	9.535	3.514	.483	.702	43.034	.4662
68	1557	5	1.221	84.532	9.546	3.513	.483	.705	43.036	.4663
69	1622	5	1.217	84.560	9.529	3.506	.479	.709	43.029	.4662
69	1641	105	1.242	84.547	9.524	3.507	.482	.698	43.012	.4662
70	1019	5	1.223	84.577	9.504	3.501	.488	.708	43.023	.4662
70	1037	105	1.241	84.587	9.499	3.486	.482	.705	42.999	.4661
71	1118	5	1.223	84.528	9.518	3.523	.492	.716	43.050	.4664
72	1144	5	1.223	84.612	9.509	3.476	.482	.698	42.997	.4660
73	1214	5	1.218	84.585	9.512	3.506	.481	.698	43.016	.4661
74	1238	5	1.270	84.527	9.509	3.505	.483	.706	43.003	.4663
74	1255	5	1.219	84.574	9.517	3.501	.486	.703	43.023	.4662
75	1314	5	1.217	84.588	9.526	3.491	.483	.695	43.011	.4661
76	1512	5	1.219	84.582	9.515	3.492	.484	.708	43.020	.4661
77	1534	5	1.216	84.522	9.550	3.528	.482	.703	43.045	.4663
78	1602	5	1.221	84.538	9.572	3.485	.486	.699	43.025	.4662
78	1621	105	1.252	84.500	9.568	3.498	.485	.697	43.017	.4663
79	1038	5	1.218	84.455	9.615	3.518	.483	.711	43.065	.4791
79	1058	5	1.204	84.561	9.529	3.520	.488	.698	43.040	.4788
79	1137	5	1.219	84.520	9.551	3.525	.484	.701	43.043	.4789
79	1155	105	1.237	84.485	9.595	3.501	.480	.702	43.032	.4790
80	1217	5	1.214	84.506	9.540	3.526	.491	.723	43.066	.4791
80	1238	105	1.235	84.523	9.557	3.502	.479	.704	43.024	.4789
81	1301	5	1.216	84.535	9.556	3.507	.482	.704	43.036	.4789
82	1325	5	1.219	84.588	9.535	3.488	.477	.693	43.005	.4786
83	1351	5	1.219	84.540	9.527	3.520	.484	.710	43.041	.4789
84	1413	5	1.225	84.355	9.636	3.576	.489	.719	43.113	.4795
84	1459	5	1.214	84.512	9.537	3.539	.484	.714	43.059	.4790
85	1516	5	1.217	84.507	9.572	3.515	.485	.704	43.047	.4790
86	1539	5	1.213	84.530	9.564	3.511	.480	.702	43.038	.4663
87	1626	5	1.215	84.509	9.559	3.532	.483	.702	43.050	.4664
88	959	5	1.216	84.549	9.548	3.510	.478	.699	43.028	.4662
88	941	105	1.238	84.543	9.537	3.499	.484	.699	43.015	.4662

Table 7.4.1. Probe Test 3 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
93	1257	5	1.223	84.527	9.552	3.519	.479	.700	43.033	.4663
93	1315	105	1.239	84.547	9.534	3.499	.482	.700	43.013	.4662
94	1333	5	1.212	84.527	9.564	3.499	.489	.709	43.045	.4789
94	1350	5	1.216	84.522	9.559	3.508	.487	.708	43.045	.4789
95	1423	5	1.217	84.509	9.546	3.511	.493	.724	43.061	.4791
96	1451	5	1.224	85.100	8.931	3.548	.486	.711	42.888	.4776
96	1511	5	1.217	84.539	9.554	3.500	.482	.708	43.034	.4789

Table 7.4.1. Probe Test 4

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG	FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	PROBE	VAPORIZER POWER, WATTS
146	2/25	111.0	.127		.23	5.6	28.	R	56.2
147	2/25	111.0	.126		.19	5.6	28.	S	61.2
148	2/25	111.0	.126		.19	5.6	28.	P	61.2
149	2/25	111.0	.125		.30	5.6	69.	S	64.4
150	2/25	111.0	.127		.26	5.6	70.	R	64.4
151	2/25	111.0	.126		.26	5.6	69.	P	64.8
152	2/25	120.0	.233		.23	4.2	71.	P	91.2
153	2/25	120.0	.231		.26	4.2	71.	S	41.0
154	2/25	120.0	.231		.26	4.2	71.	R	41.3
155	2/25	120.0	.228		.38	4.2	27.	R	45.2
156	2/25	120.0	.229		.30	4.2	27.	P	45.6
157	2/25	120.0	.229		.30	4.2	27.	S	45.6
158	2/25	120.0	.231		.30	4.2	69.	P	46.2
159	2/25	120.0	.231		.30	4.2	70.	R	42.5
160	2/25	120.0	.231		.30	4.2	70.	S	46.2
161	2/25	120.0	.229		.34	4.2	28.	S	43.2
162	2/25	120.0	.230		.34	4.2	28.	P	43.6
163	2/25	120.0	.229		.34	4.2	28.	R	43.2
164	2/25	111.0	.127		.30	5.6	70.	R	54.8
165	2/25	111.0	.126		.30	5.6	70.	S	64.0
166	2/25	111.0	.126		.34	5.6	69.	P	63.6
167	2/25	111.0	.127		.30	5.6	27.	S	63.2
168	2/25	111.0	.126		.30	5.6	27.	R	63.6
169	2/25	111.0	.126		.30	5.6	27.	P	64.0
170	2/28	111.0	.126		.34	5.6	29.	P	60.5
171	2/28	111.0	.126		.34	5.6	29.	S	60.5
172	2/28	111.0	.126		.34	5.6	29.	R	60.5
173	2/28	111.0	.127		3.79	5.6	26.	S	60.5
174	2/28	111.0	.127		3.75	5.6	28.	R	60.5
175	2/28	111.0	.127		3.79	5.6	28.	P	60.5
176	2/28	111.0	.126		.34	5.6	29.	R	60.5
177	2/28	111.0	.126		.34	5.6	29.	P	60.5
178	2/28	111.0	.126		.38	5.6	29.	S	63.2
179	2/28	111.0	.126		3.79	5.6	28.	S	60.8
180	2/28	111.0	.126		3.79	5.6	27.	P	57.8
181	2/28	111.0	.127		3.79	5.6	27.	R	58.5

Table 7.4.1. Probe Test 4 (continued)

KWD Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
146	945	5	1.214	84.616	9.538	3.486	.480	.697	43.003	.4786
147	1000	5	1.214	84.606	9.507	3.503	.479	.692	43.008	.4786
148	1018	5	1.214	84.586	9.541	3.480	.480	.700	43.012	.4787
149	1033	5	1.212	84.570	9.530	3.501	.484	.703	43.028	.4788
150	1048	5	1.213	84.637	9.492	3.479	.476	.701	42.997	.4785
151	1103	5	1.213	84.618	9.513	3.478	.478	.700	43.002	.4786
152	1134	5	1.208	84.593	9.519	3.493	.481	.706	43.021	.4661
153	1149	5	1.211	84.602	9.514	3.489	.482	.702	43.014	.4660
154	1209	5	1.206	84.559	9.543	3.500	.483	.704	43.034	.4662
154	1230	105	1.252	84.539	9.538	3.490	.480	.701	43.003	.4662
155	1250	5	1.208	84.538	9.527	3.490	.482	.705	43.022	.4661
156	1307	5	1.204	84.597	9.521	3.493	.481	.704	43.022	.4661
157	1323	5	1.209	84.572	9.531	3.500	.483	.705	43.029	.4662
158	1341	5	1.213	84.607	9.500	3.502	.479	.700	43.012	.4660
159	1356	5	1.205	84.581	9.541	3.490	.479	.704	43.024	.4661
160	1412	5	1.208	84.610	9.517	3.485	.480	.700	43.010	.4660
161	1428	5	1.206	84.624	9.509	3.482	.481	.698	43.006	.4659
162	1443	5	1.211	84.614	9.498	3.490	.482	.705	43.013	.4660
163	1518	5	1.209	84.615	9.508	3.481	.479	.708	43.011	.4660
164	1536	5	1.213	84.637	9.504	3.470	.480	.697	42.994	.4785
164	1551	105	1.236	84.536	9.533	3.518	.479	.699	43.021	.4789
165	1611	5	1.208	84.612	9.509	3.491	.480	.700	43.012	.4786
166	1632	5	1.212	84.576	9.535	3.492	.484	.702	43.023	.4787
167	1650	5	1.211	84.586	9.522	3.500	.482	.700	43.020	.4787
168	1707	5	1.210	84.604	9.525	3.481	.477	.703	43.010	.4786
169	1725	5	1.209	84.477	9.525	3.550	.499	.740	43.099	.4793
169	2740	105	1.241	84.565	9.513	3.504	.481	.696	43.005	.4788
169	1756	100	1.381	85.917	8.497	2.971	.521	.713	42.410	.4745
170	916	5	1.214	84.631	9.487	3.488	.477	.703	43.010	.4786
170	855	105	1.239	84.578	9.515	3.494	.479	.695	42.998	.4787
171	931	5	1.212	84.530	9.540	3.528	.483	.707	43.048	.4789
172	952	5	1.212	84.659	9.476	3.481	.480	.692	42.988	.4784
172	1008	5	1.210	84.658	9.480	3.485	.476	.691	42.988	.4784
173	1025	5	1.212	84.532	9.556	3.510	.485	.705	43.043	.4789
174	1044	5	1.215	84.539	9.529	3.521	.487	.709	43.045	.4789
175	1102	5	1.214	84.685	9.458	3.472	.475	.696	42.976	.4784
175	1118	5	1.212	84.664	9.434	3.494	.477	.699	42.987	.4784
176	1134	5	1.210	84.547	9.551	3.523	.479	.690	43.031	.4788
177	1154	5	1.211	84.540	9.531	3.535	.483	.700	43.044	.4789
177	1225	105	1.241	84.571	9.526	3.492	.481	.689	42.996	.4787
178	1243	5	1.213	84.596	9.538	3.479	.477	.698	43.007	.4786
179	1300	5	1.215	84.631	9.490	3.487	.479	.698	42.999	.4785
179	1316	5	1.213	84.628	9.481	3.506	.479	.693	43.003	.4786
180	1331	5	1.207	84.606	9.505	3.496	.482	.705	43.019	.4787
181	1350	5	1.211	84.635	9.502	3.483	.476	.693	42.995	.4785
181	1407	105	1.240	84.587	9.505	3.494	.481	.694	42.995	.4787

Table 7.4.1. Vaporizer Test 1

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	VAPORIZER	VAPORIZER POWER, WATTS
100	2/22	111.0	.126	7.53	5.6	25.	R	64.8
101	2/22	111.0	.126	7.57	5.6	25.	A	
102	2/22	111.0	.128	1.93	5.6	69.	A	
103	2/22	111.0	.128	3.90	2.0	25.	A	
104	2/22	111.0	.128	3.86	5.6	25.	R	64.4
105	2/22	111.0	.128	3.86	5.6	25.	A	
106	2/22	111.0	.128	7.57	2.0	25.	A	
107	2/22	111.0	.128	3.79	2.0	70.	R	14.3
108	2/22	111.0	.128	3.71	2.0	70.	R	15.6
109	2/23	120.0	.231	3.90	4.2	25.	A	
110	2/23	120.0	.229	3.82	4.2	25.	R	43.9
111	2/23	120.0	.230	7.80	1.5	25.	A	
112	2/23	120.0	.229	3.94	1.5	69.	R	22.0
113	2/23	120.0	.230	3.86	1.5	70.	A	
114	2/23	120.0	.230	3.82	4.2	70.	A	
115	2/23	120.0	.229	7.80	4.2	25.	A	
116	2/23	120.0	.228	7.83	4.2	25.	R	59.8
117	2/23	120.0	.228	3.86	1.5	25.	A	
118	2/23	111.0	.130	4.05	2.0	70.	A	
119	2/23	111.0	.130	4.01	2.0	70.	R	15.6
120	2/23	111.0	.129	7.80	2.0	25.	A	
121	2/23	111.0	.127	4.09	5.6	45.	A	
122	2/23	111.0	.128	3.97	5.6	25.	R	64.0
123	2/23	111.0	.126	3.94	5.6	25.	A	
124	2/23	111.0	.127	3.97	2.0	24.	A	
125	2/23	111.0	.126	7.65	5.6	26.	R	63.6
126	2/23	111.0	.126	7.61	5.6	25.	A	
127	2/24	120.0	.230	8.02	1.5	25.	A	
128	2/24	120.0	.233	3.82	4.2	70.	A	
129	2/24	120.0	.228	3.82	1.5	70.	R	10.2
130	2/24	120.0	.228	3.82	1.5	70.	A	
131	2/24	120.0	.229	3.90	1.5	69.	R	9.9
132	2/24	120.0	.229	7.83	4.2	25.	A	
133	2/24	120.0	.228	3.82	4.2	25.	A	
134	2/24	120.0	.227	3.86	1.5	24.	A	
135	2/24	120.0	.228	3.86	4.2	25.	R	45.2
136	2/24	120.0	.228	7.87	4.2	25.	R	46.9
137	2/24	120.0	.233	3.90	10.6	70.	A	
138	2/24	120.0	.228	3.97	4.2	70.	A	
139	2/24	120.0	.229	3.97	1.8	69.	A	
140	2/24	120.0	.228	7.83	10.8	25.	A	
141	2/24	120.0	.234	3.82	10.6	25.	A	
142	2/24	111.0	.129	3.82	10.1	25.	A	
143	2/24	111.0	.125	7.72	10.0	24.	A	
144	2/24	111.0	.130	3.86	10.2	70.	A	
145	2/24	111.0	.127	3.86	10.1	25.	A	

Table 7.4.1. Vaporizer Test 1 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
100	1118	5	1.225	84.536	9.545	3.504	.486	.705	43.032	.4789
101	1147	5	1.242	84.653	9.391	3.530	.483	.700	42.990	.4786
101	1203	5	1.240	84.631	9.408	3.537	.483	.701	43.000	.4787
101	1219	5	1.239	84.657	9.412	3.501	.482	.709	42.987	.4786
102	1239	5	1.227	84.704	9.469	3.461	.463	.676	42.941	.4782
102	1305	5	1.224	84.713	9.441	3.477	.468	.677	42.949	.4782
103	1323	5	1.209	84.216	9.661	3.714	.496	.705	43.198	.4801
103	1338	5	1.213	84.209	9.656	3.717	.500	.705	43.200	.4802
104	1359	5	1.219	84.447	9.571	3.534	.500	.729	43.090	.4793
104	1415	5	1.217	84.502	9.522	3.532	.499	.728	43.075	.4792
105	1434	5	1.275	85.237	9.144	3.262	.441	.642	42.671	.4762
105	1451	5	1.279	85.221	9.151	3.273	.441	.635	42.672	.4762
106	1510	5	1.190	83.681	10.490	3.474	.472	.693	43.275	.4809
106	1526	5	1.189	83.687	10.490	3.477	.473	.684	43.270	.4808
106	1552	105	1.247	84.507	9.530	3.534	.482	.700	43.029	.4790
107	1617	5	1.220	84.503	9.548	3.532	.488	.709	43.056	.4790
107	1634	5	1.217	84.505	9.548	3.526	.489	.715	43.059	.4791
108	1652	5	1.214	84.520	9.533	3.533	.491	.709	43.057	.4790
109	854	5	1.206	84.098	9.768	3.688	.509	.731	43.248	.4680
110	919	5	1.218	84.766	9.380	3.462	.479	.695	42.950	.4655
110	935	5	1.218	84.758	9.376	3.472	.482	.694	42.956	.4656
111	957	5	1.223	85.114	10.090	3.053	.233	.287	42.368	.4612
111	1016	5	1.220	85.046	10.084	3.100	.246	.304	42.419	.4616
112	1034	5	1.209	84.542	9.537	3.517	.486	.709	43.046	.4663
112	1055	5	1.207	84.574	9.511	3.515	.487	.706	43.037	.4662
113	1151	5	1.201	85.519	9.802	2.774	.299	.405	42.292	.4602
113	1212	5	1.203	85.529	9.786	2.776	.299	.407	42.290	.4602
114	1230	5	1.208	83.762	9.940	3.800	.527	.763	43.401	.4693
115	1248	5	1.214	84.906	9.364	3.393	.461	.663	42.865	.4648
115	1305	105	1.251	84.623	9.490	3.477	.473	.685	42.964	.4659
116	1327	5	1.217	84.514	9.514	3.540	.495	.720	43.067	.4665
117	1355	5	1.215	84.343	9.784	3.818	.367	.472	42.985	.4660
118	1420	5	1.200	83.566	9.863	3.955	.566	.852	43.576	.4831
119	1509	5	1.214	84.602	9.509	3.499	.477	.699	43.010	.4786
119	1437	105	1.236	84.583	9.512	3.492	.482	.695	43.000	.4787
120	1534	5	1.261	87.825	8.980	1.769	.128	.037	41.015	.4626
120	1550	5	1.265	87.828	9.013	1.770	.124	0.000	40.988	.4624
121	1609	5	1.209	84.562	9.572	3.502	.473	.682	43.015	.4787
122	1630	5	1.214	84.557	9.506	3.527	.487	.709	43.042	.4789
123	1649	5	1.208	84.520	9.635	3.487	.471	.679	43.020	.4787
124	1707	5	1.190	83.549	10.300	3.809	.477	.675	43.399	.4818
125	1726	5	1.214	84.457	9.466	3.635	.503	.726	43.119	.4795
125	1741	5	1.214	84.497	9.432	3.632	.505	.720	43.105	.4794
126	1801	5	1.208	84.732	9.499	3.451	.453	.657	42.927	.4780
126	1817	105	1.241	84.609	9.496	3.486	.476	.692	42.982	.4786
127	913	5	1.175	84.322	9.605	3.720	.493	.685	43.179	.4672
128	931	5	1.202	84.800	9.425	3.416	.470	.687	42.928	.4653
129	946	5	1.199	83.794	10.159	3.644	.489	.715	43.306	.4685
129	1003	5	1.199	83.786	10.159	3.655	.487	.714	43.310	.4686
130	1019	5	1.177	84.120	10.690	3.164	.356	.493	42.896	.4653

Table 7.4.1. Vaporizer Test 1 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
131	1043	5	1.207	84.322	9.500	3.690	.521	.760	43.207	.4676
131	1139	105	1.234	84.567	9.530	3.493	.479	.698	43.006	.4661
132	1201	5	1.231	84.756	9.373	3.465	.480	.696	42.946	.4656
133	1218	5	1.209	84.147	9.778	3.635	.500	.732	43.212	.4677
134	1237	5	1.205	84.077	9.520	4.204	.445	.549	43.261	.4681
135	1306	5	1.209	84.443	9.498	3.584	.517	.749	43.133	.4670
136	1328	5	1.215	84.611	9.501	3.501	.477	.695	43.006	.4660
137	1354	5	1.214	84.609	9.504	3.496	.479	.698	43.008	.4660
138	1413	5	1.224	82.574	10.579	4.179	.585	.859	43.920	.4737
139	1455	5	1.200	83.838	12.260	2.559	.143	0.000	42.397	.4620
140	1511	5	1.210	84.562	9.531	3.506	.483	.708	43.035	.4662
141	1529	5	1.218	84.579	9.510	3.500	.486	.707	43.024	.4662
142	1550	5	1.212	84.303	9.523	3.650	.526	.785	43.214	.4802
143	1607	5	1.214	84.577	9.519	3.498	.481	.711	43.026	.4788
144	1627	5	1.196	84.528	9.553	3.528	.484	.711	43.062	.4790
145	1647	5	1.240	84.379	9.573	3.587	.494	.727	43.106	.4796
145	1705	105	1.238	84.557	9.527	3.493	.482	.703	43.010	.4788

Table 7.4.1. Vaporizer Test 2

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	VAPORIZER	VAPORIZER POWER, WATTS
182	3/01	111.0	.127	3.75	10.1	33.	A	
183	3/01	111.0	.126	3.75	6.7	33.	A	
184	3/01	111.0	.124	7.53	6.6	27.	A	
185	3/01	111.0	.125	7.53	10.0	27.	A	
186	3/01	111.0	.125	3.79	6.7	28.	A	
187	3/01	111.0	.128	3.79	10.1	28.	A	
188	3/01	111.0	.126	7.57	10.0	26.	A	
189	3/01	111.0	.126	7.57	6.7	25.	A	
190	3/01	111.0	.125	7.57	6.7	26.	R	77.0

Table 7.4.1. Vaporizer Test 2 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
182	1010	5	1.243	84.596	9.479	3.491	.482	.710	43.000	.4787
183	1028	5	1.211	84.590	9.461	3.498	.496	.744	43.051	.4789
183	1051	105	1.249	84.541	9.544	3.492	.480	.694	43.001	.4788
184	1206	5	1.207	84.422	9.737	3.513	.463	.653	43.040	.4789
184	1149	105	1.248	84.517	9.543	3.509	.483	.700	43.019	.4789
185	1224	5	1.204	84.610	9.503	3.504	.481	.698	43.018	.4786
186	1252	5	1.239	84.601	9.393	3.537	.502	.728	43.035	.4789
187	1311	5	1.206	84.618	9.491	3.496	.486	.701	43.017	.4786
188	1328	5	1.206	84.309	9.628	3.590	.509	.758	43.175	.4799
188	1344	5	1.201	84.349	9.597	3.582	.509	.762	43.167	.4798
189	1359	5	1.177	84.154	9.633	3.679	.542	.815	43.314	.4808
189	1415	5	1.176	84.179	9.610	3.685	.541	.809	43.305	.4808
190	1439	5	1.214	84.569	9.530	3.508	.481	.698	43.024	.4788

Table 7.4.1. General Test 1

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG	FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	PROBE	VAPORIZER POWER, WATTS	ACCUMULATOR BYPASSED
191	4/14	123.0	.248		4.24	5.0	35.	R	59.7	NO
192	4/14	123.0	.258		4.16	5.1	34.	R	59.7	YES
193	4/14	123.0	.248		4.16	4.9	34.	R	59.3	YES
194	4/14	123.0	.254		4.01	5.1	35.	R	62.8	NO
195	4/14	123.0	.252		4.01	5.1	35.	R	62.8	YES
196	4/14	123.0	.247		4.01	5.0	35.	R	62.8	YES
197	4/14	123.0	.248		4.05	5.0	35.	R	62.4	NO
198	4/14	123.0	.245		4.05	5.0	35.	P	62.0	NO
199	4/14	123.0	.251		4.05	5.0	35.	P	62.0	YES
200	4/14	123.0	.252		4.09	5.4	34.	P	62.4	YES
201	4/14	123.0	.250		4.09	5.0	34.	P	62.0	NO
202	4/15	123.0	.250		4.13	5.0	35.	R	62.4	NO
203	4/15	123.0	.249		4.13	5.0	35.	R	62.4	YES
204	4/15	123.0	.249		4.13	5.0	36.	S	62.8	NO
205	4/15	123.0	.248		4.13	5.0	36.	S	62.4	YES
206	4/15	123.0	.248		4.24	5.0	34.	P	62.8	NO
207	4/15	123.0	.248		4.16	5.0	35.	P	62.8	YES
208	4/15	123.0	.248		4.16	5.0	35.	S	62.8	NO
209	4/18	123.0	.250		4.16	5.0	35.	R	55.5	YES
210	4/18	123.0	.250		4.24	5.0	35.	S	62.0	YES
211	4/18	123.0	.251		4.20	5.0	35.	P	59.3	YES
212	4/18	123.0	.251		4.24	5.0	35.	P	53.3	NO
213	4/19	123.0	.248		4.24	5.0	35.	R	57.0	NO
214	4/19	123.0	.248		4.24	5.0	35.	R	57.0	YES
215	4/19	123.0	.248		4.24	5.0	35.	S	57.4	YES
216	4/19	123.0	.247		4.24	5.0	35.	S	57.8	NO
217	4/19	123.0	.248		4.24	5.0	35.	P	57.8	YES
218	4/19	123.0	.248		4.24	5.0	35.	P	57.8	NO
219	4/19	123.0	.247		4.24	5.0	35.	S	57.8	NO
220	4/19	123.0	.248		4.24	5.0	35.	S	57.8	YES
221	4/19	123.0	.246		4.13	5.0	35.	P	57.8	YES
222	4/19	123.0	.247		4.13	5.0	35.	P	57.8	NO
223	4/19	123.0	.246		4.13	5.0	35.	R	57.8	YES
224	4/19	123.0	.248		4.16	5.0	35.	R	57.8	NO
225	4/19	123.0	.246		4.16	5.0	35.	P	57.8	NO
226	4/19	123.0	.248		4.13	5.0	35.	P	57.8	YES
227	4/20	123.0	.247		4.05	5.0	35.	R	57.8	YES
228	4/20	123.0	.249		4.01	5.0	35.	R	57.8	NO
229	4/20	123.0	.249		4.01	5.0	35.	S	57.8	YES
230	4/20	123.0	.249		4.01	5.0	35.	S	57.8	NO
231	4/20	123.0	.249		4.01	5.0	35.	R	57.8	YES
232	4/20	123.0	.248		4.01	5.0	35.	R	57.8	NO
233	4/20	123.0	.248		4.01	5.0	35.	P	57.8	YES
234	4/20	123.0	.246		4.01	5.0	35.	P	57.8	NO
235	4/20	123.0	.249		4.01	5.0	35.	S	57.8	YES
236	4/20	123.0	.248		4.01	5.0	35.	S	57.8	NO
237	4/20	123.0	.246		3.97	5.0	35.	S	81.8	YES
238	4/20	123.0	.246		3.97	5.0	35.	S	81.8	YES

Table 7.4.1. General Test 1 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
191	1131	6	.621	87.724	6.633	4.016	.426	.581	42.566	.4541
191	1147	6	.622	87.709	6.622	4.035	.427	.585	42.578	.4542
192	1218	6	.628	87.345	6.800	4.205	.437	.586	42.731	.4555
193	1236	6	.627	87.388	6.743	4.196	.442	.604	42.730	.4555
193	1316	106	.477	87.822	6.665	4.017	.428	.591	42.641	.4538
194	1338	6	.620	87.771	6.592	4.011	.428	.578	42.552	.4539
195	1358	6	.619	87.399	6.800	4.108	.455	.619	42.723	.4554
196	1417	6	.621	87.344	6.719	4.257	.440	.620	42.771	.4558
197	1440	6	.622	87.766	6.609	3.999	.425	.579	42.547	.4539
197	1459	106	.481	87.851	6.650	4.022	.427	.569	42.619	.4536
198	1525	6	.620	87.746	6.612	4.007	.429	.586	42.563	.4540
199	1544	6	.625	87.483	6.616	4.173	.465	.638	42.730	.4554
200	1603	6	.627	87.799	6.553	4.012	.428	.581	42.541	.4539
201	1621	6	.620	87.782	6.591	4.001	.427	.579	42.546	.4539
201	1638	106	.491	87.894	6.632	3.987	.427	.569	42.590	.4535
202	1104	6	.619	87.755	6.611	3.998	.433	.585	42.560	.4540
203	1126	6	.614	87.762	6.433	4.152	.444	.595	42.616	.4544
204	1149	6	.621	87.747	6.624	4.003	.424	.581	42.555	.4540
205	1206	6	.629	88.309	6.279	3.805	.412	.566	42.319	.4519
206	1228	6	.620	87.773	6.602	3.995	.425	.585	42.549	.4539
207	1245	6	.621	87.637	6.607	4.062	.447	.626	42.641	.4547
208	1305	6	.620	87.749	6.613	4.003	.427	.588	42.561	.4540
208	1322	6	.619	87.776	6.590	4.002	.426	.588	42.553	.4539
208	1340	106	.477	87.838	6.641	4.018	.434	.592	42.641	.4538
209	1339	6	.618	87.726	6.667	3.938	.444	.608	42.571	.4541
209	1140	106	.475	87.818	6.666	4.026	.430	.585	42.644	.4538
209	1257	106	.477	87.793	6.668	4.032	.434	.596	42.659	.4540
209	1313	106	.477	87.872	6.637	4.008	.427	.579	42.617	.4536
210	1405	6	.619	87.715	6.673	4.050	.414	.530	42.545	.4539
211	1436	6	.605	88.802	5.897	3.707	.423	.566	42.174	.4504
212	1457	6	.618	87.725	6.612	4.031	.429	.586	42.577	.4541
213	944	6	.612	87.692	6.646	4.030	.435	.585	42.592	.4542
213	909	106	.476	87.819	6.644	4.041	.432	.588	42.650	.4539
214	1000	6	.616	87.641	6.686	4.050	.431	.576	42.603	.4543
215	1016	6	.621	87.940	6.413	4.025	.421	.580	42.504	.4535
216	1034	6	.613	87.673	6.668	4.034	.430	.582	42.594	.4542
217	1050	6	.616	87.636	6.672	4.080	.430	.566	42.606	.4544
218	1109	6	.614	87.678	6.661	4.037	.428	.582	42.592	.4542
219	1127	6	.613	87.674	6.668	4.035	.428	.582	42.593	.4542
220	1142	6	.610	87.541	6.706	4.117	.435	.591	42.665	.4548
220	1259	106	.474	87.816	6.667	4.030	.426	.586	42.645	.4538
220	1315	106	.476	87.787	6.677	4.040	.432	.588	42.659	.4540
221	1331	6	.649	88.154	6.146	4.030	.432	.588	42.437	.4530
222	1348	6	.613	87.790	6.610	3.976	.425	.586	42.544	.4538
223	1404	6	.609	88.404	6.021	3.925	.445	.597	42.375	.4522
224	1422	6	.613	87.688	6.663	4.029	.427	.580	42.585	.4542
225	1437	6	.614	87.686	6.657	4.031	.431	.581	42.589	.4542
226	1453	6	.615	87.518	6.816	4.108	.406	.537	42.619	.4545
227	951	6	.630	88.048	6.333	3.961	.434	.589	42.462	.4531
228	1009	6	.611	87.669	6.655	4.047	.432	.586	42.604	.4543

Table 7.4.1. General Test 1 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
229	1025	6	.607	87.166	6.857	4.237	.485	.649	42.867	.4565
230	1054	6	.612	87.687	6.666	4.011	.435	.589	42.591	.4542
231	1113	6	.604	87.826	6.571	3.977	.433	.590	42.547	.4538
232	1130	6	.610	87.728	6.638	4.012	.432	.580	42.574	.4541
233	1145	6	.625	88.660	6.120	3.699	.385	.511	42.147	.4504
234	1213	6	.613	87.692	6.657	4.028	.427	.583	42.586	.4542
235	1229	6	.625	89.171	5.930	3.418	.372	.484	41.901	.4483
236	1245	6	.612	87.732	6.629	4.013	.433	.581	42.573	.4541
236	1031	106	.477	87.820	6.665	4.021	.433	.584	42.641	.4538
236	1419	106	.477	87.815	6.669	4.014	.435	.591	42.646	.4538
236	1434	106	.477	87.806	6.677	4.015	.435	.591	42.649	.4539
237	1450	6	.594	87.516	6.904	4.036	.406	.544	42.617	.4544
238	1508	6	.672	91.750	4.564	2.475	.238	.301	40.696	.4377

Table 7.4.1. General Test 2

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	PROBE	VAPORIZER POWER, WATTS	ACCUMULATOR BYPASSED
239	4/21	123.0	.248	3.97	5.0	35.	S	0.0	YES
240	4/21	123.0	.247	3.97	5.0	35.	S	0.0	NO
241	4/21	123.0	.246	3.97	5.0	35.	S	0.0	YES
242	4/21	123.0	.248	3.97	5.0	35.	S	0.0	NO
243	4/21	123.0	.248	3.97	5.0	35.	S	0.0	YES
244	4/21	123.0	.247	3.97	5.0	35.	S	0.0	NO
245	4/21	123.0	.248	3.97	5.0	35.	S	99.6	NO
246	4/21	123.0	.247	3.97	5.0	35.	S	98.6	YES
247	4/21	123.0	.247	3.97	5.0	35.	S	97.5	NO
248	4/21	123.0	.246	3.97	5.0	35.	S	98.1	YES
249	4/21	123.0	.247	3.97	5.0	35.	S	98.1	YES
250	4/21	123.0	.247	3.97	5.0	35.	S	98.1	NO
251	4/22	113.0	.248	4.16	5.0	35.	S	0.0	NO
252	4/22	113.0	.247	4.16	5.0	35.	S	0.0	YES
253	4/22	113.0	.248	4.16	5.0	35.	S	0.0	NO
254	4/22	113.0	.250	4.16	5.0	35.	S	0.0	YES
255	4/22	113.0	.246	4.24	5.0	35.	S	0.0	NO
256	4/22	113.0	.245	4.20	5.0	35.	S	0.0	YES
257	4/22	113.0	.245	4.13	5.0	35.	S	98.6	NO
258	4/22	113.0	.246	4.16	5.0	34.	S	97.5	YES
259	4/22	113.0	.245	4.16	5.0	35.	S	97.0	NO
260	4/22	113.0	.244	4.16	5.0	35.	S	97.0	YES
261	4/22	113.0	.245	4.16	5.0	35.	S	96.5	NO
262	4/22	113.0	.245	4.16	5.0	35.	S	96.5	YES
263	4/25	123.0	.247	5.22	5.0	35.	S	63.2	NO
264	4/25	123.0	.246	4.24	5.0	35.	S	62.0	NO

Table 7.4.1. General Test 2 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
239	1040	6	.592	85.782	7.651	4.393	.602	.980	43.564	.4623
239	903	106	.479	87.774	6.674	4.060	.431	.583	42.662	.4540
239	1025	106	.480	87.917	6.669	4.019	.427	.588	42.638	.4538
240	1056	6	.604	87.475	6.734	4.162	.442	.584	42.700	.4551
241	1119	6	.639	87.927	6.561	3.953	.394	.526	42.430	.4530
242	1134	6	.608	87.724	6.654	4.021	.421	.573	42.569	.4540
243	1149	6	.640	87.576	6.769	4.041	.418	.554	42.583	.4544
244	1205	6	.606	87.652	6.668	4.048	.436	.590	42.616	.4544
244	1326	106	.479	87.794	6.672	4.033	.432	.591	42.654	.4539
245	1342	6	.608	87.696	6.665	4.022	.428	.582	42.586	.4542
246	1357	6	.610	87.627	6.747	4.012	.424	.580	42.598	.4543
247	1415	6	.604	87.713	6.657	4.019	.427	.580	42.582	.4541
248	1431	6	.611	87.843	6.533	4.011	.425	.577	42.535	.4537
249	1453	6	.605	87.668	6.659	4.045	.431	.592	42.610	.4543
250	1508	6	.608	87.669	6.668	4.039	.434	.582	42.602	.4543
251	1151	6	.602	87.650	6.706	4.058	.422	.562	42.599	.4684
251	1132	106	.477	87.824	6.658	4.028	.431	.583	42.641	.4680
252	1207	6	.613	87.419	6.598	4.195	.472	.703	42.802	.4700
253	1222	6	.600	87.587	6.703	4.068	.441	.601	42.653	.4688
254	1242	6	.538	88.876	6.667	3.393	.246	.281	41.852	.4619
255	1301	6	.599	87.574	6.744	4.081	.430	.573	42.640	.4687
256	1318	6	.665	88.716	6.627	3.215	.329	.449	41.903	.4630
256	1334	106	.477	87.838	6.643	4.032	.430	.580	42.635	.4679
257	1359	6	.601	87.698	6.660	4.023	.430	.588	42.595	.4683
258	1419	6	.603	87.798	6.601	3.999	.427	.572	42.548	.4679
258	1436	106	.480	87.864	6.634	4.015	.430	.578	42.620	.4678
259	1519	6	.601	87.731	6.643	4.015	.427	.583	42.579	.4682
260	1502	6	.598	87.495	6.767	4.120	.436	.585	42.684	.4690
261	1559	6	.602	87.715	6.656	4.022	.427	.579	42.583	.4682
261	1617	6	.604	87.688	6.672	4.026	.430	.580	42.592	.4683
261	1634	106	.481	87.855	6.630	4.024	.429	.582	42.626	.4678
261	1651	106	.481	87.822	6.638	4.037	.434	.588	42.645	.4680
262	1536	6	.597	87.357	6.854	4.164	.439	.589	42.740	.4695
263	1153	6	.600	87.662	6.656	4.063	.433	.586	42.618	.4544
264	1425	6	.600	87.696	6.661	4.029	.431	.583	42.596	.4542

Table 7.4.1. General Test 3

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	PROBE	VAPORIZER POWER, WATTS	PROBE HEATE POWER, WATTS
265	4/25	123.0	.245	4.20	5.0	35.	S	62.0	.1
266	4/25	123.0	.246	4.20	5.0	35.	S	62.0	.4
267	4/25	123.0	.245	4.20	5.0	35.	S	62.0	1.7
268	4/25	123.0	.245	4.20	5.0	35.	S	62.0	6.7
269	4/26	123.0	.244	4.16	5.0	35.	P	62.8	0.0
270	4/26	123.0	.244	4.16	5.0	35.	P	62.0	.1
271	4/26	123.0	.244	4.16	5.0	35.	P	62.0	.4
272	4/26	123.0	.244	4.16	5.0	35.	P	62.0	1.0
273	4/26	123.0	.244	4.16	5.0	35.	P	62.0	1.7
274	4/26	123.0	.244	4.16	5.0	35.	P	62.0	3.8
275	4/26	123.0	.244	4.16	5.0	35.	P	62.0	6.7
299	4/29	123.0	.244	4.16	5.0	35.	P	62.8	0.0
300	4/29	123.0	.244	4.16	5.0	35.	P	62.4	0.0
301	4/29	123.0	.244	4.16	5.0	35.	P	62.4	0.0
302	4/29	123.0	.244	4.16	5.0	35.	P	62.4	0.0
303	4/29	123.0	.243	4.16	5.0	35.	P	62.4	0.0
304	4/29	123.0	.244	4.16	5.0	35.	P	62.4	0.0
305	4/29	123.0	.243	4.13	5.0	35.	P	62.4	0.0
306	4/29	123.0	.244	4.13	5.0	35.	P	62.4	0.0
307	4/29	123.0	.243	4.13	5.0	35.	P	62.4	0.0
308	4/29	123.0	.244	4.13	5.0	35.	P	62.4	0.0
309	4/29	123.0	.244	4.16	5.0	35.	P	62.4	0.0
310	4/29	123.0	.243	4.16	5.0	35.	P	62.4	0.0

Table 7.4.1. General Test 3 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
265	1443	6	.600	87.680	6.669	4.033	.429	.589	42.491	.4542
266	1459	6	.601	87.691	6.682	4.016	.429	.581	42.478	.4541
267	1518	6	.597	87.705	6.662	4.031	.426	.578	42.478	.4541
268	1534	6	.601	87.694	6.679	4.021	.426	.580	42.476	.4541
268	1552	106	.477	87.836	6.645	4.030	.429	.584	42.524	.4538
268	1509	106	.473	87.818	6.672	4.023	.428	.581	42.524	.4538
269	1526	6	.597	87.671	6.675	4.047	.434	.576	42.495	.4543
269	943	106	.477	87.826	6.663	4.023	.430	.582	42.524	.4538
269	959	100	.478	87.824	6.656	4.036	.429	.578	42.525	.4538
270	1048	6	.598	87.692	6.689	4.019	.427	.575	42.476	.4541
271	1104	6	.593	87.409	6.825	4.140	.444	.593	42.610	.4552
272	1125	6	.597	87.692	6.678	4.021	.429	.563	42.483	.4542
272	1143	106	.477	87.794	6.693	4.021	.433	.583	42.535	.4539
273	1159	6	.599	87.706	6.685	4.010	.427	.574	42.468	.4541
274	1217	6	.599	87.706	6.673	4.017	.426	.576	42.473	.4541
275	1233	6	.600	87.710	6.662	4.018	.432	.578	42.473	.4541
299	937	6	.590	87.595	6.690	4.088	.437	.595	42.545	.4546
299	922	106	.477	87.817	6.662	4.029	.432	.583	42.530	.4538
300	953	6	.593	87.680	6.685	4.036	.427	.582	42.495	.4542
301	1011	6	.588	87.610	6.729	4.056	.434	.583	42.524	.4545
301	1030	6	.592	87.598	6.715	4.080	.431	.584	42.530	.4545
302	1049	6	.589	87.617	6.706	4.065	.433	.590	42.527	.4545
303	1106	6	.592	87.675	6.678	4.043	.430	.583	42.497	.4543
304	1125	6	.590	87.649	6.689	4.056	.432	.584	42.511	.4544
305	1341	6	.587	87.601	6.729	4.070	.431	.582	42.529	.4545
305	1319	106	.478	87.794	6.679	4.030	.431	.589	42.539	.4539
306	1359	6	.590	87.646	6.698	4.051	.431	.584	42.510	.4544
307	1418	6	.591	87.580	6.708	4.087	.437	.597	42.549	.4547
307	1437	6	.592	87.614	6.683	4.086	.436	.590	42.534	.4546
308	1454	6	.588	87.666	6.684	4.047	.432	.584	42.505	.4543
309	1509	6	.591	87.667	6.683	4.040	.432	.587	42.503	.4543
310	1531	6	.590	87.687	6.668	4.044	.430	.581	42.494	.4542
310	1549	106	.481	87.822	6.648	4.032	.433	.584	42.528	.4538

Table 7.4.1. General Test 4

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	PROBE	VAPORIZER POWER, WATTS	NEEDLE VALVE THROTTLED
276	4/26	123.0	.244	4.16	5.0	35.	S	62.0	YES
277	4/26	123.0	.244	4.13	5.0	35.	S	62.0	YES
278	4/26	123.0	.244	4.16	5.0	35.	S	62.0	NO
279	4/26	123.0	.244	4.16	5.0	35.	S	62.0	YES
280	4/26	123.0	.244	4.16	5.0	35.	S	62.4	NO
281	4/27	123.0	.244	4.16	5.0	35.	S	62.4	NO
282	4/27	123.0	.244	4.20	5.0	35.	S	62.4	YES
283	4/27	123.0	.243	4.16	5.0	35.	S	62.4	NO
284	4/27	123.0	.243	4.16	5.0	35.	S	62.4	YES
285	4/27	123.0	.244	4.16	5.0	35.	S	62.4	NO
286	4/27	123.0	.244	4.16	5.0	35.	S	62.0	YES
287	4/27	123.0	.244	4.16	5.0	35.	S	62.4	NO
288	4/27	123.0	.243	4.16	5.0	35.	S	62.4	YES
289	4/27	123.0	.244	4.16	5.0	35.	S	62.4	NO
290	4/27	123.0	.244	4.16	5.0	35.	S	62.4	YES
291	4/27	123.0	.243	4.20	5.0	35.	S	62.4	NO
292	4/27	123.0	.243	4.20	5.0	35.	S	62.4	YES
311	5/02	123.0	.242	4.16	4.9	35.	P	62.4	NO
312	5/02	123.0	.242	4.13	4.9	35.	S	62.4	NO
313	5/02	123.0	.242	4.13	4.9	35.	S	62.4	NO
314	5/02	123.0	.243	4.13	5.0	35.	S	62.4	NO
315	5/02	123.0	.242	4.13	4.9	35.	S	62.4	NO
316	5/02	123.0	.242	4.13	4.9	35.	S	62.4	NO
317	5/02	123.0	.242	4.16	4.9	35.	S	62.8	YES
318	5/02	123.0	.242	4.13	4.9	35.	S	62.8	YES
319	5/02	123.0	.242	4.13	4.9	35.	S	62.8	YES
320	5/02	123.0	.242	4.13	4.9	35.	S	62.8	YES
321	5/02	123.0	.242	4.13	4.9	35.	S	62.8	YES

Table 7.4.1. General Test 4 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
276	1410	6	.593	87.715	6.666	4.014	.429	.579	42.471	.4541
277	1454	6	.596	87.675	6.687	4.029	.432	.582	42.491	.4542
278	1539	6	.597	87.712	6.664	4.024	.427	.576	42.473	.4541
279	1525	6	.596	87.742	6.661	4.002	.426	.571	42.457	.4539
280	1544	6	.598	87.701	6.670	4.024	.433	.574	42.478	.4541
280	1606	106	.487	87.856	6.648	3.999	.431	.580	42.502	.4536
280	1622	106	.483	87.830	6.659	4.014	.433	.581	42.518	.4538
281	956	6	.595	87.584	6.700	4.083	.441	.598	42.547	.4547
282	1011	6	.596	87.715	6.672	4.015	.427	.574	42.469	.4540
283	1026	6	.594	87.686	6.680	4.032	.429	.580	42.486	.4542
284	1041	6	.594	87.659	6.697	4.044	.426	.580	42.497	.4543
285	1056	6	.593	87.692	6.675	4.033	.427	.580	42.486	.4542
285	1312	106	.481	87.823	6.663	4.023	.431	.579	42.522	.4538
285	1327	106	.476	87.838	6.647	4.025	.433	.581	42.523	.4538
286	1342	6	.594	87.669	6.697	4.022	.428	.590	42.495	.4542
287	1401	6	.596	87.691	6.679	4.030	.425	.579	42.482	.4541
288	1416	6	.595	87.701	6.675	4.028	.428	.573	42.477	.4541
289	1435	6	.596	87.676	6.687	4.029	.431	.581	42.490	.4542
290	1450	6	.595	87.706	6.670	4.023	.425	.582	42.478	.4541
291	1510	6	.593	87.716	6.670	4.023	.426	.572	42.471	.4540
292	1527	6	.593	87.734	6.651	4.015	.427	.581	42.469	.4540
292	1543	106	.477	87.768	6.672	4.065	.432	.586	42.556	.4540
311	1046	6	.554	87.630	6.704	4.078	.437	.597	42.557	.4545
311	939	106	.478	87.806	6.677	4.028	.428	.584	42.531	.4538
311	955	106	.475	87.809	6.669	4.031	.429	.587	42.535	.4538
312	1102	6	.557	87.660	6.714	4.049	.431	.589	42.530	.4543
313	1122	6	.555	87.656	6.716	4.046	.435	.592	42.536	.4544
314	1140	6	.554	87.655	6.727	4.053	.429	.583	42.530	.4543
315	1156	6	.555	87.662	6.716	4.052	.430	.585	42.529	.4543
316	1215	6	.557	87.690	6.692	4.054	.429	.578	42.516	.4542
316	1335	106	.473	87.815	6.668	4.026	.430	.589	42.535	.4538
317	1407	6	.558	87.654	6.720	4.053	.431	.585	42.530	.4543
317	1426	6	.554	87.683	6.701	4.052	.429	.581	42.521	.4542
318	1443	6	.555	87.663	6.714	4.055	.430	.583	42.529	.4543
319	1501	6	.556	87.625	6.719	4.086	.431	.584	42.548	.4545
320	1517	6	.554	87.668	6.706	4.061	.431	.580	42.528	.4543
321	1534	6	.555	87.620	6.719	4.085	.431	.590	42.554	.4545
321	1550	6	.553	87.630	6.736	4.064	.431	.586	42.544	.4544
321	1609	106	.477	87.809	6.666	4.032	.431	.585	42.534	.4539
321	1636	106	.480	87.876	6.622	4.017	.428	.578	42.503	.4536

Table 7.4.1. General Test 5

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	PROBE	VAPORIZER POWER,WATTS	ACCUM RESIDENCE TIME,S
293	4/28	112.0	.116	1.97	12.2	56.	S	144.0	10.5
294	4/28	112.0	.116	1.93	8.6	56.	S	120.4	15.0
295	4/28	112.0	.116	1.93	5.9	56.	S	79.0	21.8
296	4/28	112.0	.117	1.89	12.2	55.	S	160.0	10.5
297	4/28	112.0	.116	1.89	8.6	55.	S	117.2	15.0
298	4/28	112.0	.117	1.93	12.2	55.	S	160.0	10.5

Table 7.4.1. General Test 5 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	Heating Value MJ/m ³	Density gm/cm ³
293	1308	6	.590	87.627	6.733	4.043	.429	.578	42.622	.4698
294	1348	6	.590	87.678	6.690	4.031	.432	.579	42.606	.4696
295	1409	6	.591	87.690	6.672	4.037	.430	.580	42.604	.4696
296	1426	6	.589	87.670	6.695	4.035	.431	.580	42.610	.4697
297	1441	6	.591	87.694	6.676	4.031	.427	.582	42.600	.4696
298	1459	6	.592	87.676	6.682	4.036	.432	.583	42.609	.4697

Table 7.4.1. General Test 6

RUN NUMBER	DATE	TEMPERATURE K	PRESSURE MPA	LNG FLOW RATE LPM	SAMPLING RATE, SLPM	DELTA P KPA	PROBE	VAPORIZER POWER, WATTS
322	5/11	123.5	.222	4.16	4.7	35.	S	62.4
323	5/11	123.5	.222	4.16	4.7	35.	S	62.4
324	5/11	123.5	.222	4.20	4.7	35.	S	62.4
325	5/11	123.5	.222	4.16	4.7	35.	S	62.4
326	5/11	123.5	.222	4.09	4.7	35.	S	99.1
327	5/11	123.5	.223	4.13	4.7	35.	S	99.1
328	5/12	121.5	.222	4.16	4.7	35.	S	99.1
329	5/12	121.5	.222	4.16	4.7	35.	S	99.1
330	5/12	121.5	.223	4.16	4.7	35.	S	99.1
331	5/12	121.5	.223	4.16	4.7	35.	S	99.1
332	5/12	121.5	.202	4.28	4.5	36.	S	62.4
333	5/12	121.5	.222	4.24	4.7	35.	S	62.4
334	5/12	121.5	.223	4.28	4.7	35.	S	62.4
335	5/16	120.0	.276	4.16	5.3	35.	S	62.8
336	5/16	120.0	.276	4.16	5.3	35.	S	62.8
337	5/16	120.0	.276	4.16	5.3	34.	P	62.8
338	5/16	120.0	.277	4.16	5.3	34.	P	62.8
339	5/17	120.0	.277	4.16	5.3	34.	P	62.8
340	5/16	120.0	.277	4.16	5.3	35.	P	62.8
341	5/16	120.0	.274	4.16	5.3	35.	P	62.8

Table 7.4.1. General Test 6 (continued)

Run Number	Time	Mix	N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	C ₆ +	CO ₂	O ₂	Heating Value MJ/m ³	Density gm/cm ³
322	1120	7	.129	84.280	10.850	3.419	.326	.744	.125	.1140	0.000	.008	.005	43.931	.461
323	1147	7	.127	84.307	10.841	3.397	.325	.742	.124	.1110	0.000	.009	.017	43.905	.461
324	1214	7	.127	84.274	10.841	3.424	.328	.746	.123	.1110	0.000	.009	.017	43.925	.461
325	1306	7	.133	84.331	10.830	3.401	.324	.737	.122	.1120	0.000	.007	.003	43.901	.461
326	1511	7	.140	84.319	10.831	3.390	.324	.736	.124	.1130	0.000	.007	.015	43.891	.461
327	1553	7	.134	84.328	10.843	3.387	.323	.736	.123	.1120	0.000	0.000	.014	43.894	.461
328	1106	8	.721	83.855	10.781	3.335	.322	.730	.122	.1100	0.000	.006	.018	43.613	.465
329	1134	8	.721	83.857	10.780	3.335	.321	.732	.122	.1110	0.000	.006	.015	43.615	.465
330	1220	8	.721	83.849	10.790	3.336	.321	.731	.122	.1120	0.000	.004	.014	43.620	.465
331	1256	8	.709	83.851	10.790	3.338	.322	.732	.125	.1130	0.000	.007	.013	43.632	.465
332	1442	8	.720	83.819	10.800	3.348	.321	.734	.123	.1130	0.000	.006	.016	43.633	.465
333	1507	8	.715	83.852	10.799	3.333	.322	.727	.122	.1110	0.000	.007	.012	43.619	.465
334	1558	8	.712	83.856	10.781	3.343	.322	.731	.120	.1120	.001	.005	.017	43.624	.465
334	1623	8	.713	83.864	10.789	3.339	.322	.724	.123	.1110	0.000	0.000	.016	43.620	.465
334	1648	100	1.382	85.968	8.489	2.939	.517	.705	0.000	0.0000	0.000	0.000	0.000	42.380	.459
334	1713	100	1.379	85.953	8.503	2.950	.518	.697	0.000	0.0000	0.000	0.000	0.000	42.385	.459
335	1115	9	2.373	82.427	10.571	3.320	.315	.731	.122	.1130	.002	.010	.016	42.921	.471
336	1139	9	2.366	82.363	10.600	3.336	.319	.742	.125	.1210	0.000	.011	.016	42.962	.471
337	1222	9	2.369	82.388	10.590	3.322	.316	.734	.123	.1330	0.000	.011	.014	42.953	.471
338	1252	9	2.365	82.342	10.590	3.323	.315	.728	.122	.1120	.007	.009	.006	43.050	.471
339	1327	9	2.361	82.363	10.580	3.313	.314	.729	.125	.1140	.007	.008	.005	43.050	.471
339	1352	9	2.362	82.363	10.590	3.309	.313	.728	.124	.1110	.073	.012	.015	43.018	.471
340	1512	9	2.375	82.361	10.619	3.328	.314	.732	.122	.1140	.015	.006	.015	42.959	.471
341	1541	9	2.376	82.369	10.610	3.328	.318	.734	.121	.1160	0.000	.009	.019	42.939	.471

7.4.2. Confirmation Test Data

Table 7.4.2 lists the raw data for shipboard tests. The analyses showing zero butanes content are boil off gas samples and have been omitted from all statistical evaluations. Table 7.4.3 lists the second flow facility test data. The runs corresponding to each test were:

Test A	Runs 1 through 17
Test B	Runs 18 through 39
Test C	Runs 40 through 44
Test D	Runs 45 through 58
Test E	Runs 59 through 62
Test F	Runs 63 through 71
Test G	Runs 72 through 77. The heating values are

real gas values computed using the A.G.A. method [1]; liquid densities were computed using the extended corresponding states method [3]. Two sets of heating values and specific gravities are given for the flow facility tests. The values listed with the operating variables are measured while those listed with the composition are computed from the given composition.

Table 7.4.2. Shipboard Test Data

RUN NUMBER	DATE	TIME	SAMPLE	SAMPLE RATE SLPM	HEADER PRESS MPA	PANEL PRESS MPA	LNG TEMP K	VAPORIZER OUTLET TEMP °C
1	6/12	1655	L	17.500	.381	.232	114.6	-10.0
2	6/12	1705	G	.567	.352	.108	114.6	-10.0
3	6/12	1740	L	13.900	.421	.219	114.6	-30.0
4	6/12	1750	L	21.600	.432	.253	114.6	-30.0
5	6/12	1755	G	.587	.432	.112	114.6	-30.0
6	6/12	1855	L	8.900	.312	.205	114.6	10.0
7	6/12	1855	L	8.900	.312	.205	114.6	10.0
8	6/12	1920	G	.067	.492	.115	114.6	10.0
9	6/12	1925	L	23.900	.492	.267	114.6	10.0
10	6/12	1925	L	23.900	.492	.267	114.6	10.0
12	6/13	1715	L	12.500	.552	.294	114.6	10.0
13	6/13	1715	G	1.043	.552	.115	114.6	10.0
14	6/13	2100	L	13.900	.521	.363	114.6	10.0
15	6/13	2100	G	1.043	.521	.115	114.6	10.0
16	6/13	2215	L	14.900	.521	.253	114.6	10.0
17	6/13	2215	L	14.900	.521	.253	114.6	10.0
18	6/13	2220	G	.587	.521	.112	114.6	12.0
19	6/13	2300	L	3.400	.521	.287	114.6	12.0
20	6/13	2300	G	.151	.521	.115	114.6	14.0
21	6/13	2350	L	32.300	.512	.329	114.6	14.0
22	6/13	2350	G	.488	.512	.112	114.6	15.0
23	6/14	245	L	18.200	.481	.191	114.6	15.0
24	6/14	245	G	.924	.481	.112	114.6	15.0
25	6/14	400	L	24.900	.481	.315	114.6	15.0
26	6/14	410	L	25.700	.481	.308	114.6	15.0
27	6/14	410	G	1.023	.481	.112	114.6	15.0
28	6/14	445	L	24.200	.472	.274	114.6	15.0
29	6/14	450	G	1.023	.472	.112	114.6	15.0
30	6/17	1140	L	19.400	.421	.267	114.6	10.0
31	6/17	1142	L	19.400	.421	.267	114.6	10.0
32	6/17	1145	G	.567	.421	.110	114.6	10.0
33	6/17	1200	L	22.000	.412	.239	114.6	10.0
34	6/17	1205	G	1.439	.412	.110	114.6	15.0
35	6/17	1210	L	14.100	.421	.225	114.6	15.0
36	6/17	1220	G	.567	.412	.110	114.6	10.0

Table 7.4.2. Shipboard Test Data (continued)

RUN NUMBER	DATE	TIME	SAMPLE	SAMPLE RATE SLPM	HEADER PRESS MPA	PANEL PRESS MPA	LNG TEMP K	VAPORIZER OUTLET TEMP °C
37	6/17	1230	L	13.000	.412	.212	114.6	10.0
38	6/17	1235	G	1.221	.412	.110	114.6	20.0
39	6/17	1240	L	18.500	.452	.205	114.6	20.0
40	6/18	1455	L	18.200	.421	.225	115.0	20.0
41	6/18	1500	G	.468	.101	.105	115.0	20.0
42	6/18	1520	L	17.900	.432	.205	115.0	20.0
43	6/18	1550	L	19.100	.501	.232	115.0	20.0
44	6/18	1555	G	.330	.512	.106	115.0	20.0
45	6/18	1605	L	21.300	.532	.239	115.0	20.0
46	6/18	1610	G	.429	.532	.107	115.0	20.0
47	6/18	1625	L	21.000	.532	.239	115.0	20.0
48	6/18	1640	L	25.600	.532	.253	115.0	20.0
49	6/18	1640	G	.389	.532	.108	115.0	20.0
50	6/18	1720	G	.468	.521	.108	115.0	20.0
51	6/18	1722	L	20.100	.521	.232	115.0	20.0
52	6/18	1735	L	22.300	.521	.219	115.0	20.0
53	6/21	1708	L	20.400	.412	.225	116.0	0.0
54	6/21	1720	G	.587	.452	.112	116.0	50.0
55	6/21	1720	L	24.300	.452	.239	116.0	0.0
56	6/21	1730	G	1.677	.352	.112	116.0	25.0
57	6/21	1735	L	6.200	.352	.225	116.0	0.0
58	6/22	2110	G	.785	.501	.108	116.5	25.0
59	6/22	2114	L	23.300	.501	.294	116.5	0.0
60	6/22	2125	L	18.300	.501	.253	116.5	0.0
61	6/22	2230	G	1.003	.501	.108	116.5	25.0
62	6/22	2255	L	24.300	.501	.239	116.5	0.0
63	6/22	2305	G	1.003	.501	.108	116.5	25.0
64	6/22	2310	L	24.300	.501	.239	116.5	0.0
65	6/22	2325	L	24.300	.501	.239	116.5	0.0
66	6/22	2330	G	1.003	.501	.108	116.5	25.0
67	6/22	2345	L	25.900	.501	.239	116.5	25.0
68	6/22	2400	L	29.000	.501	.212	116.5	25.0

Table 7.4.2. Shipboard Test Data (continued)

Run Number	Time	Composition, mol %							Heating Value MJ/m ³	Density gm/cm ³
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	C ₅ ⁺		
1	1655	.114	83.827	11.565	3.457	.542	.496	0.000	43.883	.4751
2	1705	9.724	74.976	10.943	3.337	.533	.487	0.000	39.977	.4987
3	1740	.119	84.090	11.291	3.432	.560	.507	0.000	43.813	.4745
4	1750	.105	83.994	11.535	3.360	.527	.480	0.000	43.796	.4743
5	1755	.993	83.300	11.575	3.214	.479	.441	0.000	43.314	.4756
6	1855	.413	90.401	6.762	1.934	.225	.220	.044	41.077	.4526
7	1855	.409	90.401	6.781	1.940	.225	.219	.026	41.064	.4525
8	1920	.209	83.762	11.553	3.442	.541	.494	0.000	43.833	.4752
9	1925	.125	84.142	11.286	3.378	.540	.495	.036	43.791	.4743
10	1925	.123	84.196	11.224	3.383	.542	.493	.040	43.781	.4742
12	1625	2.879	97.080	.041	0.000	0.000	0.000	0.000	36.694	.4262
12	1715	.118	85.200	10.511	3.161	.507	.457	.046	43.401	.4710
13	1715	.111	85.139	10.564	3.173	.510	.461	.042	43.426	.4712
14	2100	.110	85.177	10.545	3.163	.507	.457	.040	43.408	.4710
15	2100	.093	85.025	10.681	3.189	.511	.459	.042	43.475	.4715
16	2215	.108	85.140	10.574	3.174	.508	.457	.040	43.423	.4711
18	2200	.112	85.096	10.574	3.184	.525	.466	.042	43.453	.4714
19	2300	.113	85.243	10.642	3.069	.464	.424	.044	43.322	.4704
20	2300	.110	85.071	10.591	3.197	.521	.460	.052	43.467	.4715
21	2344	2.751	96.080	.935	.234	0.000	0.000	0.000	37.131	.4298
21	2344	2.729	96.096	.935	.240	0.000	0.000	0.000	37.143	.4298
21	2350	.110	85.141	10.555	3.179	.514	.459	.042	43.429	.4712
22	2350	.110	85.170	10.529	3.172	.516	.461	.042	43.421	.4711
23	245	.108	85.124	10.557	3.175	.516	.465	.054	43.450	.4713
24	245	.108	85.114	10.566	3.186	.511	.459	.056	43.451	.4713
25	400	.109	84.791	10.981	3.123	.502	.451	.044	43.504	.4719
26	410	.110	85.090	10.577	3.200	.514	.452	.046	43.455	.4714
27	410	.109	85.108	10.574	3.191	.515	.458	.046	43.447	.4713
28	445	.110	85.169	10.517	3.181	.513	.451	.050	43.429	.4712
29	450	.116	85.172	10.508	3.183	.511	.459	.052	43.424	.4712
30	1140	.029	82.571	12.317	3.850	.625	.560	.048	44.534	.4798
31	1142	.013	82.312	12.564	3.865	.628	.565	.054	44.631	.4805
32	1145	.003	82.409	12.428	3.925	.621	.552	.052	44.624	.4804
33	1200	0.000	82.511	12.396	3.897	.628	.568	0.000	44.549	.4798
34	1205	.029	82.394	12.361	3.979	.631	.562	.044	44.622	.4805

Table 7.4.2. Shipboard Test Data (continued)

Run Number	Time	Composition, mol %							Heating Value MJ/m ³	Density gm/cm ³
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	C ₅ +		
35	1210	.002	81.390	13.540	3.835	.615	.559	.060	44.888	.4828
36	1220	.030	82.773	12.303	3.751	.573	.526	.046	44.396	.4788
37	1230	.031	82.525	12.374	3.847	.617	.561	.046	44.538	.4799
38	1235	0.000	81.456	13.350	3.938	.631	.572	.052	44.911	.4828
39	1240	0.000	82.569	12.325	3.859	.622	.562	.064	44.568	.4799
39	1430	.164	95.191	4.570	.075	0.000	0.000	0.000	39.060	.4327
39	1609	.168	93.311	6.446	.075	0.000	0.000	0.000	39.595	.4382
39	1918	.353	96.626	2.926	.095	0.000	0.000	0.000	38.530	.4285
40	1611	.004	79.094	14.497	4.831	.795	.714	.066	46.020	.4909
42	1712	0.000	79.095	14.489	4.838	.797	.716	.064	46.027	.4909
43	1803	0.000	79.184	14.426	4.817	.799	.714	.062	45.991	.4907
43	2147	0.000	79.128	14.459	4.830	.800	.713	.070	46.019	.4909
44	1836	.002	79.132	14.443	4.842	.797	.716	.068	46.018	.4909
45	2116	0.000	79.049	14.498	4.864	.803	.720	.066	46.054	.4911
46	2147	0.000	79.027	14.511	4.868	.801	.724	.070	46.064	.4912
47	1622	0.000	78.955	14.572	4.871	.804	.724	.074	46.092	.4914
48	1654	.002	78.920	14.592	4.887	.808	.724	.068	46.102	.4915
49	1727	0.000	78.884	14.625	4.900	.805	.725	.062	46.111	.4916
50	1759	.003	78.839	14.539	4.863	.803	.719	.234	46.254	.4926
51	1828	0.000	78.041	15.548	4.830	.792	.717	.072	46.331	.4935
51	2037	0.000	79.048	14.516	4.857	.789	.722	.068	46.047	.4911
51	2218	0.000	78.990	14.550	4.872	.802	.723	.064	46.071	.4913
52	1858	0.000	78.988	14.550	4.871	.806	.721	.064	46.074	.4913
53	1744	0.000	78.310	15.091	4.993	.808	.725	.074	46.314	.4919
54	1819	.003	78.215	15.165	5.051	.793	.715	.058	46.328	.4921
55	2110	0.000	78.407	15.019	4.993	.806	.713	.062	46.268	.4916
55	1852	0.000	78.378	15.034	4.996	.808	.725	.060	46.283	.4917
55	2213	0.000	78.372	15.025	5.016	.809	.718	.060	46.288	.4917
56	2038	0.000	78.344	15.113	4.960	.806	.712	.066	46.279	.4917
57	2143	.003	78.595	14.923	4.950	.764	.699	.066	46.173	.4909
57	2242	.005	78.607	14.921	4.942	.775	.692	.056	46.160	.4909
58	1147	0.000	78.675	14.809	4.926	.790	.718	.084	46.184	.4903
59	1330	0.000	77.984	15.292	5.086	.820	.732	.088	46.456	.4924
59	1400	0.000	77.996	15.282	5.092	.814	.741	.074	46.445	.4923
60	1450	0.000	77.931	15.343	5.096	.818	.741	.070	46.463	.4924

Table 7.4.2. Shipboard Test Data (continued)

Run Number	Time	Composition, mol %							Heating Value MJ/m ³	Density gm/cm ³
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	C ₅ +		
60	2145	0.000	77.929	15.356	5.100	.815	.724	.076	46.459	.4924
61	2150	0.000	77.912	15.347	5.131	.817	.726	.066	46.467	.4925
62	2220	0.000	77.857	15.375	5.135	.824	.735	.074	46.499	.4927
62	2252	0.000	77.877	15.425	5.145	.823	.730	0.000	46.430	.4922
62	2113	0.000	77.894	15.360	5.122	.822	.734	.068	46.478	.4926
63	2324	0.000	77.953	15.329	5.103	.808	.727	.080	46.454	.4924
64	1034	0.000	77.820	15.401	5.136	.824	.743	.076	46.516	.4928
64	1502	0.000	78.125	15.199	5.066	.808	.733	.068	46.388	.4919
64	1859	0.000	77.773	15.487	5.120	.828	.728	.064	46.508	.4928
64	2043	0.000	77.909	15.349	5.113	.830	.728	.072	46.475	.4925
65	1719	0.000	77.869	15.376	5.121	.820	.731	.084	46.496	.4927
66	2301	0.000	78.324	15.431	5.164	.355	.727	0.000	46.043	.4896
67	1757	0.000	77.874	15.380	5.110	.827	.730	.080	46.490	.4926
68	1828	0.000	77.825	15.428	5.130	.818	.731	.066	46.495	.4927

Table 7.4.2. LNG Flow Facility Test 2 Data.

RUN NUMBER	DATE	TIME	VAPORIZER NUMBER	TEST SECTION PRESS,MPA	PANEL PRESS MPA	SAMPLING TEMPERATURE RATE,SLPM	TEMPERATURE K	HEATING VALUE MJ/M	SPECIFIC GRAVITY
1	3/09	1025	1	.342	.249	12.26	124.4	43.192	.650
2	3/09	1025	2	.342	.256	13.56	124.4	43.192	.650
3	8/09	1100	1	.344	.257	12.42	124.4	43.192	.650
4	8/09	1100	2	.344	.244	12.64	124.4	43.192	.650
5	8/09	1320	1	.459	.307	16.59	125.2	43.211	.650
6	8/09	1525	1	.459	.232	18.19	125.8	43.211	.650
7	8/09	1525	2	.459	.218	18.53	125.8	43.211	.650
8	8/09	1600	1	.451	.265	19.00	125.2	43.192	.650
10	8/09	1615	1	.451	.251	19.14	125.4	43.192	.650
11	3/09	1615	2	.451	.230	15.13	125.4	43.192	.650
12	3/09	1630	1	.441	.248	19.42	124.3	43.192	.650
13	8/09	1630	2	.441	.220	13.43	124.3	43.192	.650
14	8/09	1645	1	.443	.243	19.14	124.7	43.192	.650
15	8/09	1645	2	.443	.222	13.43	124.7	43.192	.650
16	8/09	1700	1	.449	.256	19.42	124.8	43.192	.650
17	8/09	1700	2	.449	.228	15.13	124.8	43.192	.650
18	8/10	1030	2	.484	.291	18.29	132.7	43.528	.658
19	8/10	1100	2	.487	.266	20.92	132.6	43.546	.658
20	8/10	1130	2	.476	.249	21.74	131.9	43.546	.658
21	8/10	1145	2	.497	.284	22.33	133.1	43.546	.658
22	8/10	1200	2	.493	.293	22.68	132.8	43.546	.658
23	8/10	1220	2	.481	.281	22.68	132.4	43.546	.658
24	8/10	1230	2	.481	.281	21.54	132.4	43.528	.658
25	8/10	1245	2	.481	.302	22.68	132.4	43.528	.658
26	8/10	1300	2	.487	.287	22.68	132.8	43.528	.658
27	8/10	1330	2	.479	.272	21.26	132.5	43.546	.658
28	8/10	1400	2	.480	.280	22.68	132.5	43.546	.658
29	8/10	1415	2	.474	.274	22.68	132.4	43.546	.658
30	8/10	1430	2	.492	.285	22.39	133.1	43.546	.658
31	3/10	1445	2	.489	.283	22.39	133.0	43.546	.658
32	8/10	1530	2	.522	.336	13.49	134.5	43.621	.660
33	8/10	1545	2	.481	.288	12.94	132.3	43.677	.660
34	8/10	1600	2	.481	.288	12.94	132.1	43.639	.658
35	3/10	1615	2	.493	.320	13.81	132.6	43.639	.658
36	8/10	1630	2	.489	.310	13.65	132.5	43.639	.658
37	8/10	1645	2	.486	.300	13.45	132.4	43.639	.658
38	8/10	1700	2	.491	.298	13.29	132.7	43.639	.658
39	3/10	1715	2	.489	.303	13.45	132.6	43.639	.658
40	8/11	1140	2	.494	.280	19.44	122.8	44.254	.690
41	8/11	1155	2	.435	.255	16.03	118.6	43.863	.680
42	8/11	1210	2	.413	.247	16.03	115.7	43.695	.680
43	3/11	1225	2	.498	.291	16.03	122.8	43.733	.680
44	8/11	1245	2	.481	.254	15.09	121.8	43.733	.680
45	8/11	1400	2	.468	.233	9.06	122.3	43.844	.680
46	8/11	1400	1	.468	.254	9.60	122.3	43.844	.680

Table 7.4.2. LNG Flow Facility Test 2 Data (continued).

RUN NUMBER	DATE	TIME	VAPORIZER NUMBER	TEST SECTION PRESS,MPA	PANEL PRESS MPA	SAMPLING TEMPERATURE RATE,SLPM	K	HEATING VALUE MJ/M	SPECIFIC GRAVITY
47	8/11	1430	2	.485	.264	9.69	123.4	43.826	.680
48	8/11	1430	1	.485	.264	9.47	123.4	43.826	.680
49	8/11	1500	2	.477	.257	9.69	122.7	43.826	.680
50	8/11	1500	1	.477	.264	9.60	122.7	43.826	.680
51	8/11	1530	2	.478	.250	9.69	123.5	43.882	.680
52	8/11	1530	1	.478	.264	10.62	123.5	43.882	.680
53	8/11	1645	2	.453	.212	5.16	121.1	44.124	.690
54	8/11	1645	1	.453	.267	6.03	121.1	44.124	.690
55	8/11	1700	2	.437	.189	5.00	120.1	44.124	.680
56	8/11	1700	1	.437	.210	5.95	120.1	44.124	.680
57	8/11	1731	2	.454	.220	4.58	121.6	44.180	.682
58	8/11	1731	1	.454	.240	5.86	121.6	44.180	.682
59	8/12	1420	1	.463	.460	18.95	117.3	44.049	.717
60	8/12	1435	1	.459	.448	18.95	116.8	44.049	.717
61	8/12	1445	1	.483	.480	19.38	118.3	44.049	.717
62	8/12	1455	1	.462	.452	19.09	117.0	44.049	.717
63	8/12	1505	1	.451	.216	16.67	115.9	44.049	.717
64	8/12	1505	3	.451	.216	15.81	115.9	44.049	.717
65	8/12	1515	3	.450	.229	15.13	115.9	44.031	.717
66	8/12	1525	1	.467	.226	16.83	117.6	44.031	.717
67	8/12	1525	3	.467	.229	15.94	117.6	44.031	.717
68	8/12	1535	3	.469	.234	15.81	117.8	44.031	.717
69	8/12	1545	1	.461	.220	16.83	117.5	44.068	.717
70	8/12	1545	3	.461	.220	15.13	117.5	44.068	.717
71	8/12	1555	3	.454	.212	15.13	116.4	44.068	.717
72	8/12	1650	1	.473	.238	17.79	117.5	44.198	.716
73	8/12	1650	3	.473	.218	15.13	117.5	44.198	.716
74	8/12	1705	1	.477	.222	16.35	117.9	44.198	.716
75	8/12	1715	1	.468	.213	16.35	117.5	44.198	.715
76	8/12	1715	1	.408	.248	17.20	117.5	44.198	.715
77	8/12	1740	3	.445	.210	14.64	115.8	44.198	.715

Table 7.4.2. LNG Flow Facility Test 2 Data (continued).

Run Number	Integrator	Composition, mol %									Heating Value MJ/m ³	Specific Gravity
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	C ₆ +		
1	A	0.000	85.017	11.991	2.414	.223	.188	.037	.048	.083	43.147	.647
2	A	0.000	84.827	12.109	2.424	.225	.188	.038	.047	.082	43.205	.648
2	B	.000	84.839	12.139	2.431	.220	.189	.035	.047	.092	43.207	.648
3	A	0.000	85.399	11.079	2.347	.221	.186	.037	.040	.083	43.018	.645
3	B	0.000	85.460	11.647	2.344	.216	.185	.030	.046	.072	42.974	.644
4	A	0.000	83.228	13.424	2.701	.252	.211	.042	.052	.090	43.788	.658
4	B	0.000	84.934	12.090	2.433	.223	.191	.034	.049	.077	43.179	.648
5	A	0.000	84.575	12.340	2.480	.233	.195	.036	.049	.084	43.306	.650
5	B	0.000	84.560	12.367	2.483	.227	.195	.035	.050	.093	43.305	.651
6	A	0.000	85.070	11.968	2.383	.226	.187	.037	.047	.082	43.123	.647
6	B	.006	85.065	11.954	2.397	.221	.188	.031	.047	.091	43.129	.647
7	A	0.000	84.843	12.319	2.482	.217	.186	.034	.044	.076	43.255	.649
7	A	0.000	84.672	12.299	2.473	.218	.185	.033	.044	.076	43.244	.649
7	B	0.000	84.686	12.262	2.481	.211	.185	.031	.045	.079	43.241	.649
8	A	0.000	84.346	12.531	2.514	.236	.196	.039	.050	.087	43.389	.651
8	B	0.000	84.726	12.263	2.470	.215	.186	.030	.045	.065	43.211	.648
8	B	0.000	84.375	12.522	2.508	.231	.199	.034	.050	.080	43.366	.651
10	A	0.000	83.763	13.007	2.539	.246	.205	.041	.051	.089	43.596	.655
10	B	0.000	83.775	12.987	2.608	.241	.206	.034	.054	.094	43.595	.655
11	A	0.000	84.759	12.305	2.379	.217	.184	.036	.044	.070	43.194	.648
11	B	0.000	84.680	12.278	2.456	.221	.193	.033	.050	.090	43.265	.649
12	A	.000	84.911	12.073	2.425	.227	.189	.039	.048	.083	43.182	.648
12	B	0.000	84.943	12.054	2.422	.221	.192	.031	.050	.087	43.174	.647
13	A	0.000	84.695	12.260	2.469	.223	.190	.036	.047	.081	43.253	.649
13	A	0.000	84.684	12.268	2.470	.224	.191	.037	.046	.080	43.256	.649
13	B	0.000	84.728	12.232	2.475	.222	.192	.032	.044	.075	43.233	.648
13	B	0.000	84.697	12.247	2.474	.222	.192	.031	.047	.090	43.262	.649
14	A	0.000	84.911	12.080	2.427	.227	.189	.037	.047	.082	43.183	.648
14	B	0.000	84.955	12.054	2.419	.224	.191	.034	.048	.076	43.159	.647
15	A	0.000	85.065	11.903	2.428	.235	.195	.039	.049	.086	43.155	.647
15	B	0.000	85.006	11.896	2.430	.230	.194	.037	.049	.096	43.166	.647
16	A	0.000	84.656	12.285	2.464	.231	.193	.038	.049	.084	43.277	.649
16	B	0.000	84.685	12.266	2.458	.226	.192	.033	.047	.094	43.269	.649
17	A	0.000	84.606	12.262	2.537	.225	.192	.036	.045	.076	43.295	.650
17	B	0.000	84.642	12.259	2.532	.222	.193	.031	.046	.075	43.275	.649
18	A	0.000	83.916	12.804	2.575	.241	.204	.057	.052	.091	43.559	.654
18	B	0.000	83.960	12.855	2.582	.240	.204	.037	.043	.079	43.508	.653
19	A	0.000	83.946	12.850	2.573	.238	.200	.056	.050	.087	43.538	.654
19	B	.007	83.938	12.848	2.562	.230	.202	.037	.042	.075	43.473	.653
20	A	0.000	83.849	12.908	2.597	.245	.204	.056	.052	.090	43.584	.654
20	B	0.000	83.906	12.895	2.586	.236	.206	.036	.047	.080	43.537	.654
21	A	0.000	84.148	12.877	2.410	.224	.187	.055	.036	.062	43.375	.651
21	B	0.000	84.190	12.855	2.402	.217	.186	.034	.042	.075	43.360	.651
22	A	0.000	83.914	12.838	2.595	.240	.205	.058	.053	.092	43.571	.654
22	B	.004	83.970	12.839	2.582	.239	.204	.035	.047	.080	43.556	.653
23	A	0.000	83.909	12.878	2.569	.242	.203	.059	.052	.090	43.558	.654
23	B	.006	83.946	12.877	2.566	.238	.205	.037	.046	.081	43.508	.653
24	A	0.000	83.855	12.932	2.574	.240	.202	.057	.051	.089	43.571	.654
24	A	.005	83.822	12.947	2.589	.238	.202	.057	.052	.090	43.592	.654
24	B	.005	83.895	12.939	2.574	.234	.202	.035	.044	.072	43.506	.653

Table 7.4.2. LNG Flow Facility Test 2 Data (continued).

Run Number	Integrator	Composition, mol %									Heating Value MJ/m ³	Specific Gravity
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	C ₆ +		
24	B	.005	83.883	12.941	2.578	.238	.201	.036	.046	.074	43.518	.653
25	A	.000	83.917	12.912	2.548	.240	.201	.055	.051	.087	43.543	.654
25	B	.011	83.931	12.902	2.562	.236	.203	.036	.044	.074	43.495	.653
26	A	.000	83.865	12.904	2.584	.243	.203	.058	.053	.091	43.578	.654
26	B	.000	83.931	12.887	2.571	.240	.204	.037	.047	.084	43.522	.653
27	A	.000	83.774	12.964	2.612	.245	.205	.058	.053	.091	43.613	.655
27	B	.000	83.835	12.961	2.602	.240	.207	.037	.045	.074	43.546	.654
28	A	.000	83.834	12.921	2.598	.243	.204	.059	.052	.090	43.589	.655
28	B	.000	83.834	12.924	2.587	.240	.206	.035	.046	.070	43.517	.653
29	A	.007	83.808	12.940	2.596	.245	.205	.057	.052	.090	43.592	.655
29	B	.009	83.856	12.947	2.596	.236	.203	.034	.046	.073	43.525	.654
30	A	.000	83.857	12.920	2.586	.240	.202	.057	.051	.088	43.572	.654
30	B	.000	83.918	12.907	2.587	.237	.204	.034	.042	.071	43.506	.653
31	A	.007	83.822	12.921	2.600	.245	.204	.060	.052	.090	43.591	.655
31	B	.013	83.881	12.913	2.588	.239	.205	.036	.046	.079	43.525	.654
32	A	.000	83.020	13.549	2.746	.257	.216	.061	.055	.096	43.891	.660
32	A	.000	83.028	13.552	2.735	.254	.216	.062	.056	.097	43.887	.660
32	B	.000	83.134	13.529	2.732	.250	.216	.040	.050	.079	43.816	.658
32	B	.006	83.063	13.548	2.737	.252	.216	.035	.049	.093	43.840	.659
33	A	.014	83.715	13.008	2.607	.246	.205	.059	.053	.092	43.623	.655
33	B	.021	83.765	12.997	2.612	.237	.207	.035	.045	.081	43.561	.654
34	A	.007	83.714	13.030	2.600	.245	.207	.056	.052	.090	43.620	.655
34	B	.009	83.750	13.015	2.618	.240	.205	.032	.043	.089	43.581	.654
35	A	.000	83.678	13.036	2.630	.246	.206	.059	.053	.091	43.649	.656
35	B	.013	83.733	13.021	2.619	.242	.208	.035	.046	.078	43.576	.654
36	A	.006	83.770	12.968	2.605	.245	.204	.059	.052	.090	43.608	.655
36	B	.000	83.840	12.965	2.602	.239	.205	.037	.047	.066	43.534	.654
37	A	.008	83.840	12.915	2.591	.243	.205	.057	.052	.090	43.579	.654
37	B	.006	83.876	12.922	2.593	.240	.205	.033	.047	.078	43.530	.654
38	A	.000	83.738	12.960	2.594	.243	.205	.057	.052	.091	43.600	.655
38	B	.006	83.845	12.935	2.607	.241	.207	.036	.044	.081	43.549	.654
39	A	.011	83.890	12.860	2.597	.243	.203	.057	.051	.088	43.560	.654
39	B	.014	83.946	12.853	2.579	.238	.204	.037	.042	.088	43.511	.653
40	A	1.239	80.334	14.747	2.966	.276	.234	.044	.059	.102	43.915	.673
40	B	1.608	80.059	14.684	2.958	.274	.234	.040	.053	.090	43.722	.674
41	A	1.978	79.784	14.591	2.931	.279	.233	.044	.059	.101	43.571	.675
41	B	1.627	80.086	14.633	2.952	.276	.235	.044	.052	.096	43.711	.674
42	A	1.275	80.325	14.771	2.921	.276	.231	.044	.058	.100	43.876	.673
42	B	1.643	80.064	14.681	2.939	.270	.236	.040	.049	.083	43.675	.673
43	A	1.240	80.366	14.712	2.961	.282	.234	.045	.059	.102	43.908	.673
43	B	1.596	80.103	14.642	2.961	.274	.235	.042	.051	.095	43.725	.674
44	A	1.914	79.523	14.872	2.973	.282	.234	.044	.058	.100	43.701	.677
44	B	1.581	79.818	14.921	2.988	.277	.234	.044	.052	.087	43.817	.675
45	A	1.176	80.352	14.779	2.973	.282	.236	.044	.059	.101	43.957	.673
45	B	1.506	80.116	14.729	2.961	.274	.234	.043	.050	.088	43.771	.674
46	A	1.902	79.803	14.645	2.935	.280	.232	.044	.058	.101	43.616	.675
46	B	1.570	80.107	14.679	2.951	.276	.235	.044	.052	.088	43.733	.674
47	A	1.159	80.222	14.912	2.972	.285	.237	.046	.062	.107	44.019	.674
47	B	1.488	79.967	14.858	2.980	.279	.235	.045	.053	.097	43.850	.675
48	A	1.204	80.340	14.796	2.940	.281	.235	.044	.059	.102	43.933	.673

Table 7.4.2. LNG Flow Facility Test 2 Data (continued).

Run Number	Integrator	Composition, mol %									Heating Value MJ/m ³	Specific Gravity
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	C ₆ ⁺		
48	A	1.202	50.307	14.798	2.975	.278	.234	.045	.059	.102	43.953	.674
48	B	1.544	50.079	14.723	2.956	.276	.235	.042	.052	.092	43.764	.674
48	B	1.547	50.071	14.727	2.962	.275	.233	.044	.054	.087	43.760	.674
49	A	1.170	50.278	14.842	2.975	.283	.235	.046	.061	.106	43.988	.674
49	B	1.511	50.052	14.780	2.963	.276	.235	.041	.056	.087	43.790	.674
50	A	1.215	50.291	14.800	2.973	.282	.234	.044	.059	.102	43.949	.674
50	B	1.573	50.054	14.733	2.958	.274	.235	.039	.052	.083	43.736	.674
51	A	1.128	50.521	14.719	2.923	.277	.230	.044	.058	.100	43.919	.672
51	B	1.458	50.278	14.604	2.922	.271	.232	.040	.050	.085	43.737	.673
52	A	1.191	50.291	14.842	2.949	.264	.236	.044	.060	.103	43.963	.674
52	B	1.534	50.022	14.777	2.907	.273	.238	.044	.053	.088	43.789	.674
53	A	.819	79.584	15.717	3.124	.296	.246	.046	.061	.106	44.492	.679
53	B	1.056	79.436	15.668	3.113	.288	.244	.044	.056	.095	44.336	.679
54	A	.906	80.445	14.907	3.008	.287	.239	.045	.060	.104	44.131	.673
54	B	1.181	80.245	14.858	3.000	.278	.238	.043	.052	.099	43.994	.674
55	A	.884	80.742	14.805	2.874	.272	.225	.043	.057	.098	43.994	.671
55	B	1.157	80.557	14.702	2.803	.268	.227	.036	.048	.080	43.826	.671
56	A	.907	79.901	15.344	3.095	.295	.245	.046	.061	.106	44.322	.677
56	B	1.933	79.668	14.737	2.971	.277	.235	.040	.053	.086	43.619	.676
57	A	.857	79.880	15.482	3.044	.288	.239	.045	.060	.105	44.337	.676
57	B	.861	79.920	15.462	3.027	.283	.239	.044	.060	.103	44.312	.676
57	B	2.173	79.399	14.831	2.915	.270	.229	.043	.052	.090	43.518	.676
57	B	2.018	79.598	14.800	2.909	.269	.229	.039	.052	.086	43.555	.675
58	A	.938	79.998	15.260	3.058	.292	.242	.047	.061	.105	44.259	.676
58	B	1.217	79.798	15.202	3.058	.286	.242	.044	.053	.101	44.112	.676
59	A	4.114	74.164	17.153	3.670	.343	.291	.054	.078	.134	44.102	.707
59	B	4.009	74.247	17.150	3.665	.340	.292	.050	.066	.121	44.076	.706
60	A	4.132	74.144	17.160	3.664	.348	.291	.054	.076	.132	44.093	.707
60	B	5.573	73.601	16.478	3.520	.329	.280	.046	.065	.106	43.185	.707
61	A	4.123	74.140	17.181	3.654	.348	.291	.055	.076	.132	44.099	.707
61	B	4.571	74.707	16.405	3.506	.328	.276	.047	.063	.097	43.514	.702
62	A	4.118	74.165	17.152	3.660	.343	.292	.055	.077	.134	44.100	.707
62	B	4.081	74.238	17.158	3.661	.343	.293	.046	.064	.116	44.062	.706
63	A	4.159	74.116	17.156	3.663	.348	.291	.055	.078	.134	44.087	.707
63	B	4.854	74.355	16.449	3.515	.328	.280	.049	.065	.104	43.443	.704
64	A	4.053	74.242	17.150	3.650	.349	.292	.055	.077	.133	44.117	.707
64	B	4.482	74.608	16.536	3.531	.329	.281	.053	.065	.116	43.642	.703
65	A	4.083	74.271	17.097	3.648	.346	.291	.055	.077	.133	44.086	.706
65	B	4.037	74.352	17.090	3.646	.343	.291	.055	.064	.116	44.059	.705
66	A	4.125	74.012	17.269	3.686	.349	.293	.054	.078	.134	44.148	.708
67	A	4.106	74.143	17.100	3.689	.348	.291	.054	.077	.133	44.119	.707
67	A	4.112	74.169	17.155	3.666	.343	.291	.055	.077	.133	44.099	.707
67	B	4.049	74.256	17.156	3.666	.343	.289	.049	.065	.127	44.092	.706
67	B	4.063	74.236	17.169	3.667	.344	.291	.053	.066	.112	44.076	.706
68	A	4.065	74.203	17.195	3.634	.347	.291	.054	.077	.134	44.115	.707
68	B	4.008	74.274	17.183	3.668	.343	.292	.053	.069	.111	44.104	.706
69	A	4.132	74.121	17.182	3.658	.350	.292	.055	.077	.134	44.103	.707
69	B	4.092	74.194	17.187	3.672	.343	.290	.051	.066	.115	44.074	.706
70	A	4.034	74.214	17.191	3.655	.349	.292	.054	.077	.134	44.139	.707
70	B	4.002	74.530	17.248	3.685	.305	.290	.051	.070	.121	43.860	.702

Table 7.4.2. LNG Flow Facility Test 2 Data (continued).

Run Number	Inte- grator	Composition, mol %									Heating Value MJ/m ³	Specific Gravity
		N ₂	CH ₄	C ₂ H ₆	C ₃ H ₈	iC ₄ H ₁₀	nC ₄ H ₁₀	iC ₅ H ₁₂	nC ₅ H ₁₂	C ₆ ⁺		
71	A	4.035	74.196	17.194	3.669	.349	.292	.055	.077	.134	44.149	.707
71	B	3.985	74.289	17.189	3.670	.340	.292	.049	.066	.121	44.121	.706
72	A	3.562	74.420	17.384	3.713	.356	.297	.056	.078	.135	44.423	.707
72	A	3.558	74.411	17.399	3.720	.348	.296	.055	.076	.136	44.425	.707
72	B	3.528	74.480	17.406	3.713	.347	.296	.053	.068	.109	44.378	.706
72	B	3.517	74.479	17.407	3.720	.349	.297	.050	.069	.113	44.392	.706
73	A	3.991	74.209	17.227	3.665	.349	.292	.055	.078	.135	44.175	.707
73	B	3.955	74.284	17.217	3.660	.344	.292	.052	.067	.109	44.136	.706
74	A	3.226	74.647	17.493	3.712	.355	.296	.055	.079	.136	44.582	.706
74	B	3.187	74.716	17.486	3.729	.349	.296	.053	.068	.117	44.553	.705
75	A	2.878	74.726	17.712	3.749	.360	.302	.056	.080	.138	44.810	.706
75	B	3.384	75.315	16.849	3.610	.335	.285	.050	.065	.108	44.187	.701
76	A	3.259	74.596	17.481	3.742	.355	.296	.057	.077	.134	44.580	.706
76	B	3.220	74.673	17.482	3.733	.349	.297	.056	.069	.122	44.554	.705
77	A	3.980	74.310	17.178	3.627	.347	.292	.054	.077	.134	44.139	.706
77	B	4.661	74.669	16.365	3.493	.326	.278	.047	.061	.100	43.464	.702

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